

Worker and Environmental Assessment of Potential Unbound Engineered Nanoparticle Releases

Phase I Final Report: Data Collection

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1.0

Introduction

At the Lawrence Berkeley National Laboratory (LBNL), there are approximately sixty laboratories where unbound engineered nanoparticles (UNP) are used.¹ Each of these laboratories need to be evaluated for worker exposures and environmental releases of UNP. To obtain information regarding potential releases of UNP, LBNL retained RJ Lee Group, Inc., to conduct a multi-phase pilot study on a subset of LBNL laboratories where UNP are used.

The goals of the pilot study are to comply with Department of Energy (DOE) Notice N456.1, *The Safe Handling of Unbound Engineered Nanoparticles*, Contractor Requirements Document (DOE 2009), as follows:

- Conduct a worker and environmental exposure assessment for the potential release of UNP.
- Assess the need for appropriate controls for worker and environmental protection (including the use of high-efficiency particulate air [HEPA] exhaust filtration to protect the environment).
- Establish a worker and environmental monitoring program (air and water) for UNP based on preliminary exposure assessment and guidance provided in the DOE Nanoscale Science Research Centers approach document.

In addition, the pilot study is designed to meet the requirements of the DOE Nanoscale Science Research Centers *Approach to Nanoscale ES&H* including Attachment 1, Example Industrial Hygiene Sampling Protocol (DOE 2008).

The pilot study also supports LBNL's commitment to integrated safety management (ISM) by addressing several of the core functions of ISM, including hazard and risk analysis, establishment of controls, and providing analysis and feedback for continuous improvement.

The pilot study will be performed in the following four phases to review and assess the ESH-related hazards and controls of UNP research activities conducted by LBNL's Environmental Energy Technologies Division (EETD):

Phase I: Review and gather data

Phase II: Develop preliminary control bands

Phase III: Validate and modify control bands

Phase IV: Establish a periodic monitoring and assessment program

This report provides the results of the Phase I investigation. It also includes background information on the regulatory and technical challenges, and discusses the approach taken to assign controls in the pilot study.

¹ As of January 2009. The number of laboratories where UNP is used is not static and has probably increased since January 2009.

2.0

Background

Engineered nanoparticles are defined by DOE as “intentionally created . . . particle[s] with one or more dimensions greater than 1 nm and less than 100 nm.” The DOE defines UNP as “engineered nanoparticles that . . . are not contained within a matrix that would be expected to prevent the nanoparticles from being separately mobile and a potential source of exposure.” Additional detail is provided in DOE Notice N456.1 (DOE 2009).

Nanotechnology and the use of UNP is a rapidly developing field. At this time there are no regulatory environmental release limits or worker exposure limits for nanomaterials. Some consensus standards have been issued, but they are still under development by committees such as ASTM Committee E56 on Nanotechnology² and the ANSI-accredited U.S. Technical Advisory Group to ISO/TC 229.³

The DOE Office of Science, in support of ISM, has provided some guidance (DOE 2008) and a policy statement (DOE 2009). The policy specifically states the following:

DOE and its contractors will identify and manage potential health and safety hazards and potential environmental impacts at sites through the use of existing Integrated Safety Management Systems, including Environmental Management Systems. DOE organizations working with nanomaterials will stay abreast of current research and guidance relating to the potential hazards and impacts of nanomaterials, and will ensure that this best current knowledge is reflected in the identification and control of these potential hazards and impacts at their facilities.

This pilot study is an effort to satisfy the DOE policy and to address the uncertainties inherent in a rapidly developing technology. It is also designed to support the LBNL research organizations by addressing the five ISM core functions. A control banding approach is being used to provide guidance on risk management of UNP.

Control banding is a method for summarizing risks and controls, especially for hazards that are not currently well-characterized, as is the case for most nanomaterials (Zalk and Nelson 2008). The control banding approach is discussed in numerous environment, safety, and health (ESH) publications and is used by the pharmaceutical industry and by the European Union countries for most ESH hazards (Money 2003; Farris, Ader, and Ku 2006; Naumann 1996). For nanotechnology and nanomaterials specifically, control banding has been addressed in papers by staff at Lawrence Livermore National Laboratory (LLNL), and their work was used in developing and tailoring control bands for the LBNL project (Zalk and Nelson 2008, Paik et al. 2009).

² <http://www.astm.org/COMMIT/COMMITTEE/E56.htm>

³ http://www.ansi.org/standards_activities/standards_boards_panels/tc229.aspx?menuid=3

In general, the control banding process can be described by simple formulas, which are similar for addressing worker and environmental risks:

Health Hazard + Exposure Potential → Worker Risk → Worker Exposure Control

Environmental Hazard + Release Potential → Environmental Risk → Environmental Control

When the control is commensurate with the risk, the hazard is successfully mitigated. Studies indicate that control banding is highly successful at determining adequate controls when validated by subsequent professional evaluations and workplace monitoring, such as those that will be performed in Phase III of the LBNL pilot study (Hashimoto 2007).

In Phase I of the pilot study, information was gathered and UNPs were analyzed to determine each research activity's potential health/environmental hazard and the exposure/release potential. Health and environmental hazards are used to develop hazard bands that range from little hazard (for known, relatively safe materials) to extreme hazard (materials that are highly toxic or have unknown toxicity). If health and environmental hazards are unknown, hazard bands can still be developed where there is sufficient information available to determine the exposure and release potential. In general, toxicity, reactivity, and fire hazards for a given material increase as particle size decreases. Surface area for a given mass also increases as particle size decreases.

Information on health/environmental hazards and exposure/release potential will be used to develop control bands. Control bands can point to appropriate controls for new processes and, for the LBNL pilot study, be used to determine the adequacy of already established controls documented in research-specific activity hazard documents and observed during Phase I evaluations of the processes.

3.0

Approach

UNP activities performed under the direction of eight LBNL principal investigators were initially identified within EETD and were selected for the pilot study. However, a ninth principal investigator was identified during the Phase I investigation and included in the study. The EETD was chosen for this study because the various research programs using UNPs in EETD reasonably represent the range of materials, activities, and hazards expected throughout LBNL in general. In addition, it was determined that eight to ten research programs could be adequately addressed in a pilot study.

The EETD pilot study includes the following principal investigators and laboratory locations.

1. John Kerr, Building 62, Lab 246
2. Thomas Richardson, Building 62, Lab 342
3. Vincent Battaglia, Building 70, Labs 295/297/299 and 206
4. Gao Liu, Building 70, Lab 226
5. Robert Kostecki, Building 70, Lab 108
6. Samuel Mao, Building 70, Lab 163
7. Rick Russo, Building 70, Lab 157
8. Don Lucas, Building 70, Labs 291/293
9. Andre Anders, Building 70, Lab 274

To understand health/environmental hazards and exposure/release potential, EETD principal investigators and/or their research colleagues were interviewed and observed while demonstrating their use of nanomaterials. In addition, RJ Lee Group personnel obtained and analyzed samples of nanomaterials currently used in the research (researchers' starting [bulk] materials). A list of the samples collected as part of the Phase I study is provided in Appendix A.

Prior to the laboratory inspection, documents related to the various processes involving EETD UNP research activities were reviewed to provide background information on the research performed at each location. The laboratories were inspected by Gary Casuccio and Randall Ogle of RJ Lee Group on June 9, 10, and 11, 2009. Gary and Randy were accompanied by Linnea Wahl (Pilot Study Project Manager) throughout the inspection process. Guy Kelley (EETD Safety Coordinator) and Tim Roberts (EETD ESH Liaison and Industrial Hygienist) of LBNL were also present for most of the laboratory inspections. The principal investigators or their associates provided an overview of the processes and demonstrated the types of work performed using various nanomaterials, including UNP. This inspection provided information that can be used to make a preliminary assessment as to whether there exists the potential for worker exposure or environmental release of UNP.

During the walk-through of the laboratories, a TSI Condensation Particle Counter (CPC) Model 3007 was employed to provide preliminary data on background (not process-related) levels (particles/cm³) of

particles in the size range between 10 nm and $>1\text{ }\mu\text{m}$ (particle size must be greater than 10 nm in diameter and the upper cutoff for particles counted is estimated somewhere between 1–10 μm). This instrument provides only particle count data (no information on particle chemistry).

The following information on hazard and exposure attributes was gathered in Phase I.

1. particle size
2. particle morphology
3. elemental chemistry
4. solubility
5. toxicities of starting materials
6. amount of material used
7. dustiness (or potential for the material to become airborne based on the task/process)
8. number of people doing the work
9. duration of the operation
10. frequency of the operation

4.0

Results

As part of the Phase I pilot study, numerous researchers and support staff were interviewed in an effort to understand the pertinent aspects of work hazards and controls. In all cases, the researchers were forthcoming and demonstrated a working understanding of the ISM processes as related to their research. In general, researchers had clearly thought through the ESH aspects of their work (in accordance with the ISM core functions of research and work planning, hazard identification, risk analysis, and application of hazard controls). It was noted that work performance was commensurate with the established controls.

Additionally this project helps satisfy the last of the ISM core functions, analysis and feedback, by reviewing the work performance and discussing the hazards and controls with researchers and their management. Researchers had worked with their LBNL safety support and subject matter experts in planning, hazard identification, workplace monitoring, and performance of work, and there was an excellent line of communication established between researchers and ESH personnel. The LBNL ESH personnel involved in this project were well versed on the nature of hazards associated with nanotechnology research.

The types of nanomaterials used by EETD researchers include lithium (metal) salts, silica and silicon, carbonaceous materials, and gold and silver metals. Solid and powdered dry materials are used, as well as nanoparticles in liquid suspensions and nondispersible nanoparticles bound to substrate surfaces. Presently, regulations for many carbonaceous UNP materials, such as carbon nanotubes, have not been promulgated. Some carbonaceous materials, such as carbon black (and acetylene black), are not currently regarded as UNP but are subject to other governmental standards and regulations. Graphene, a carbonaceous material that has only one dimension less than 100 nm, is also not currently considered as UNP.

In general, all EETD research with nanomaterials evaluated in this pilot study presented similar hazards and exposure attributes as observed during Phase I. The attribute information gathered in Phase I is summarized in Table 1; details are provided in the following discussions of specific projects. In Phase II, attribute information will be used to identify health/environmental hazards and exposure/release potentials, and to develop control bands for most of the research reviewed in Phase I. The exceptions are work under Gao Liu and Andre Anders, which were determined to be unlikely sources of occupational exposure or environmental releases of UNP by any release route and thus will not be evaluated further in this pilot study.

Table 1 General Summary of Risk Attributes

	Risk Attribute	Summary Result
1	Particle size	Particle sizes ranged from 1 nm to on the order of micrometers; all materials comprised primarily nanoparticles (i.e., 2 dimensions <100 nm), with exception of lithium titanate, lithium iron phosphate, and graphene
2	Particle morphology	<ul style="list-style-type: none"> • Carbon black, acetylene black, fumed silica, and silicon samples had aciniform characteristics with rounded and irregular primary particles • Au and Ag materials were rounded, angular, and rod-shaped • Lithium titanate particles were irregular • Lithium iron phosphate were primarily rounded and rod-shaped • Iron disulfide were irregular and in droplet form
3	Elemental chemistry	Specific to the material; see detailed discussion of specific project
4	Solubility	All materials are insoluble in water
5	Toxicity	Low to high/unknown
6	Amount of material used	Nanogram to gram quantities
7	Dustiness/airborne potential	<ul style="list-style-type: none"> • Dry material is potentially prone to being dusty (the silica and silicon materials appear to be most prone to airborne release) • Carbon materials in paste are less dusty • UNP in solutions are not considered dusty unless they become dried
8	Number of people doing the work	1–3 staff members
9	Duration of the operation	Use of UNP less than 60 minutes/process
10	Frequency of the operation	Infrequent

A summary of the particle count results obtained during the Phase I evaluation study is provided in Table 2. The vast majority of the particles counted at Buildings 62 and 70 are most likely introduced into laboratories from the building air supply (outside environment). It should be noted that there are many non-research sources of nanomaterials in ambient air. Based on the CPC data, results indicate that LBNL laboratories have a much lower concentration of particles than a local hotel.

Results of analyses of samples of starting materials used by EETD researchers are provided in Appendix B. These data will be used in Phases II and III to develop and validate control bands for EETD activities. However, it was noted in Phase I that a high level of control is already in use for laboratories evaluated in this project. All activities with the potential for airborne releases were performed in fume hoods or glove boxes, with the exception of the use of graphene (not considered a UNP at this time). None of these control devices had high-efficiency particulate air (HEPA) filters, so the potential for environmental release from building ventilation exhaust will be evaluated further in Phase III of this project.

Table 2 Summary of Condensation Particle Counter Results

Location	Sampling Results (particles/cm ³ of air sampled)
Doubletree Hotel guest room	18K – 25K
Bldg 62, Lab 246	4.5K – 5K
Bldg 62, Lab 342	5K – 7K; no increase in counts in fume hood
Bldg 70, Conference Room, 1 st Floor	2K – 4K; during evacuation drill 3K
Bldg 70, Lab 295/297/299	~1.5K
Bldg 70, Lab 163	2.5K – 3K
Bldg 70, Lab 157	1.5K – 2K
Bldg 70, Lab 291/293	6K – 8K
Bldg 70, Lab 247	2K

Prudent laboratory practices were followed in each laboratory. Researchers wore lab coats, safety glasses, and polymer gloves when working with potential UNP materials. EETD staff were aware that potential nanomaterial waste must be managed in compliance with LBNL's system for managing and disposing of these materials as hazardous waste. Liquids that may contain nanomaterials are bottled, contained, and labeled as required by LBNL rules. LBNL staff were aware that sinks and drains must not be used to dispose of liquids that could contain UNP. Solid waste with potential nanoparticulates are managed through the LBNL-wide hazardous waste system. Laboratory interviews and observations indicated LBNL rules are being followed. Barring accidental or malicious activities, environmental releases of nanomaterials from building drain systems are not credible. Similarly, all solid hazardous waste is managed and this system addresses proper management of nanomaterials. In Phase I, there were no sources of liquid or solid nano-pollution requiring further analysis.

The following sections provide details of the research programs evaluated in this study.

4.1 John Kerr: Building 62, Lab 246

4.1.1 Battery Materials Program

John Kerr and his staff conduct research on battery materials in Building 62, Lab 246. Nanomaterials used in the process include fumed silica and carbon (acetylene) black. Although lithium-based compounds are used in these processes, they are typically in the millimeter-size range and not a nano-size material of interest in this study.

During the inspection of Lab 246, only the process involving fumed silica was evaluated. The process involving carbon (acetylene) black in the glove box was not demonstrated since it is similar to that used by Thomas Richardson, which was scheduled to be evaluated in Building 62 Lab 342.

4.1.2 Process Overview: Fumed Silica

The work with the fumed silica is performed in an approved, functioning fume hood and inside an inert atmosphere glove box. The process consists of transferring a portion of the fumed silica from the original chemical container and placing it in a labeled glass jar using a spatula. The transfer is done inside the fume hood. The closed secondary container is then placed inside the glove box where the silica is transferred to a mixing vessel and is mixed into a paste with an appropriate solvent. Various forms of carbon black or acetylene black may also be added to the paste.

Photos illustrating the process involving fumed silica are provided in Appendix C.

4.1.3 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 3, based on discussions with the researcher, observations of the process, analysis of the starting materials (fumed silica and carbon [acetylene] black), and review of reference documents. A summary of the scanning electron microscopy using energy-dispersive x-ray spectroscopy (SEM/EDS) performed on fumed silica and carbon (acetylene) black samples are provided in Appendix B. Inductively coupled plasma optical emission spectroscopy (ICP-OES) results for fumed silica are also provided in Appendix B.

For work in the fume hood (which is functional), appropriate work practices are followed and appear to be adequate for worker protection. Thus, no significant occupational exposure would be anticipated. Once the material is made into a paste, the risk of release of particles is greatly reduced. The highest probability for environmental airborne release occurs through the fume hood (the exhaust is not filtered) when the fumed silica is transferred into the glass jar via a spatula. Any loss of UNP would be expected to be small based on duration of this activity (less than a 5-minute process that occurs 1–3 times a week). The significance of the presence of fumed silica in the exhaust air will be studied further in Phase III of this pilot study.

Table 3 Specific Risk Attributes Associated with John Kerr's UNP Research

Risk Attribute	Building 62 Lab 246 Fumed Silica	Building 62 Lab 246 Carbon (Acetylene) Black
Particle size	Primary particles ~20–30 nm; agglomerated structures on the order of micrometers	Primary particles ~30–40 nm; agglomerated structures on the order of micrometers
Particle morphology	Aciniform structures comprising rounded particles	Aciniform structures comprising rounded and irregularly shaped particles
Elemental chemistry	SEM/EDS: Si, O; trace: Cl, K, Na, Ca, S, F ICP-OES: Major: Si; trace: Na, P, B	SEM/EDS: C
Solubility (water)	Insoluble	Insoluble
Toxicity	High	Medium
Amount of material used	< 10 g	< 10 g
Dustiness/airborne potential	High	Moderate
Number of people doing the work	1–3	1–3
Duration of the operation	< 5 min	< 5 min
Frequency of the operation	1–3 x/week	1–3 x/week

For work in the glove box, loss of UNP into the exhaust air is unlikely, but should also be quantified through further study. Although the practice of exhausting the glove box vacuum system into the building exhaust ventilation system is good and greatly reduces the potential for occupational exposures, further study is suggested. There is little documented ESH information on the movement of nanoparticles from the glove box environment through the vacuum system and subsequent release into the ventilation system and the outside environment.

4.2 Thomas Richardson: Building 62, Lab 342

4.2.1 Battery Materials Program

Research conducted in this laboratory is directed by Thomas Richardson and focuses on battery materials and graphene. Nanomaterials being utilized in these research areas include graphene, gold, silver, and carbon (acetylene) black.

4.2.2 Process Overview: Graphene/Gold

The process was demonstrated by Ki-Joon (KJ) Jeon, working in an approved, functioning fume hood. The graphene membranes used in the demonstration were synthesized previously (not observed during Phase I) using the substrate-free gas-phase method. The process involves placing a small amount of graphene (contained in a glass jar) via a spatula into a small glass vial containing ethanol. The graphene/ethanol solution is ultrasonically agitated to get the graphene into suspension.

Gold nanoparticles (BBI Inc., 10 nm average diameter) were introduced into the suspension using a hand pipette. The gold/graphene/ethanol solution was then shaken by hand for approximately 30 seconds to form a dispersion of nanoparticles and graphene. A drop of the suspension was deposited onto a copper transmission electron microscopy (TEM) grid with a lacey carbon support, which was air-dried leaving the graphene coated with gold particles attached to the TEM grid. The entire process is performed in less than 5 minutes and occurs 1–2 times a month. Although not demonstrated, the researcher indicated that the process involving the use of Ag is identical to that demonstrated with Au.

Photos illustrating the graphene/gold process are provided in Appendix D.

4.2.3 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 4, based on discussions with the researcher, observations of the process, analysis of the starting materials (nanogold, nanosilver, and graphene), and review of reference documents. A summary of the analyses performed on nanogold, nanosilver, and graphene samples are provided in Appendix B.

Evaluation of the process indicates there is minimal opportunity for worker exposure or environmental airborne release of UNP. Current work practices appear to be adequate for worker protection. Work is performed in the fume hood (which is functional) and appropriate work practices are followed. The greatest opportunity for particle release (barring accidental release through breakage of the glass jars) occurs when the graphene is placed in the ethanol via a spatula or when the gold/graphene/ethanol solution is pipetted and dried on the TEM grid. Any environmental loss of UNP would be through the fume hood (the exhaust is not filtered) and would be expected to be small based on duration of this activity (the process takes less than 5–10 minutes to perform and occurs 1–2 times per month). In Phase III, samples will be collected in the fume hood to document whether worker exposure or environmental release of graphene or nanogold particles occurs during this process.

Table 4 Specific Risk Attributes Associated with Thomas Richardson's UNP Research

Risk Attribute	Building 62 Lab 342 Gold	Building 62 Lab 342 Silver	Building 62 Lab 342 Graphene	Building 62 Lab 342 Carbon Black	Building 62 Lab 342 Acetylene Black
Particle size	Average 3 nm in diameter, ranging from 1.4–3 nm	Rounded and angular averaged <100 nm, ranging from ~10 nm to 150 nm; rod-shaped were 50–75 nm in diameter and >500 nm in length	Size information was difficult to determine; individual platelets appear on the order of 200 nm	Primary particles ~50–75 nm; agglomerated structures on the order of micrometers	Primary particles ~30–40 nm; agglomerated structures on the order of micrometers
Particle morphology	Rounded particles, often spherical; also observed in clusters	Rounded, angular, and rod-shaped particles; agglomerates observed	Agglomerated slender platelets; unable to distinguish individual sheets	Aciniform structures comprising agglomerated irregular, angular, and rounded particles	Aciniform structures comprising irregularly shaped primarily particles
Elemental chemistry	SEM/EDS: Au	SEM/EDS: Ag with Si residue	SEM/EDS: C trace; O Si	SEM/EDS: C	SEM/EDS: C
Solubility (water)	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
Toxicity	High	High	Low	Medium	Medium
Amount of material used	< 10 ng	< 10 ng	< 100 µg	< 10 mg	< 10 µg
Dustiness/airborne potential	Low	Low	Moderate	Moderate	Moderate
Number of people doing the work	1	1	1	1–3	1–3
Duration of the operation	< 5 min	< 5 min	< 10 min	< 30 min	< 30 min
Frequency of the operation	1–2 x/month	1–2 x/month	1–2 x/month	1–3 x/week	1–3 x/week

It should be noted that graphene (as used in the manner of this research) is not currently considered a UNP since only one dimension (thickness) would typically have the potential to be less than 100 nm. Most current definitions require two or more dimensions of less than 100 nm to be classified as a nanoparticle. Complicating this issue is DOE Notice 456.1, which defines a nanoparticle as having only one dimension less than 100 nm (DOE 2009). Thus, the standard definitions of nanoparticle are in flux and there is some conflict. It is suggested that this issue can be better addressed in Phase III or IV of the pilot study when graphene analysis results and specific morphological information are reviewed.

Results

If graphene is designated as a UNP, further administrative actions and additional studies may be required to evaluate the potential for release in the ventilation exhaust. Although graphene may fall under the definition of a nanoparticle, it should only have one dimension in the nano-size range, which lowers the risk of target organ exposure and toxicity. Further study on the morphology of graphene will be conducted specifically to determine if more than one dimension is less than 100 nm. If graphene particulate is present with two dimensions less than 100 nm, then nano-ESH rules will apply to its use.

4.2.4 Electroactive Polymer Program

Research in this laboratory is being conducted on electroactive polymers to reduce the tendency of lithium batteries to overcharge. Nanoparticles of interest in this research include carbon (acetylene) black. Other materials are used (such as titanium disulfide and lithium iron phosphate) but they are not nano-size materials.

4.2.5 Process Overview: Carbon (Acetylene) Black

This process involves coating TiS_2 electrodes with a slurry consisting of TiS_2 powder, carbon (acetylene) black, and poly(vinylidenedifluoride) in n-methylpyrrolidinone, and is performed inside an inert-atmosphere glove box, as demonstrated by Thomas Richardson. A small portion of carbon (acetylene) black is placed, via a spatula, in a plastic weighing dish positioned on a scale. After weighing the carbon (acetylene) black, it is placed in a glass jar and mixed into a slurry by adding a solvent via a hand pipette. An active ingredient such as LiFePO_4 is also added to the slurry (typically in the millimeter size and not a nano-size material). The slurry is then placed on a cathode using a wood stirring rod. Photos illustrating the process are provided in Appendix E.

4.2.6 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 4, based on discussions with the researcher, observations of the process, analysis of the starting materials (carbon black and acetylene black), and review of reference documents. A summary of the analyses performed on carbon and acetylene black samples are provided in Appendix B.

Because the entire process is performed inside a glove box, there appears to be little opportunity for any worker exposure to UNP. The loss of UNP from the glove box into the exhaust air is also unlikely, but should be quantified through further study. Although the practice of exhausting the glove box vacuum system into the building exhaust ventilation system is good, and greatly reduces the potential for occupational exposures, further study is suggested. There is little documented ESH information on the movement of nanoparticles from the glove box environment through the vacuum system, and the subsequent release into the ventilation system and the outside environment.

4.3 Vincent Battaglia: Building 70, Labs 295/297/299

4.3.1 Battery Fabrication Program

Research conducted in these laboratories, referred to as the Battery Fabrication Laboratory, is directed by Vincent Battaglia and focuses on battery materials including high-voltage, high-energy cell fabrication and materials characterization. With respect to nanomaterials, areas of research include carbon (acetylene) black, silicon powder, lithium titanate powder, and lithium iron phosphate powder.

4.3.2 Process Overview: Carbon (Acetylene) Black/Lithium Compounds

Paul Ridgway provided an overview of the research conducted in these laboratories, and Honghe Zheng and Xiangyun Song provided demonstrations of the various processes performed. Battery fabrication research is performed inside inert-atmosphere glove boxes, where lithium-titanium active materials are combined with carbon black and a solvent to make a slurry or paste. The paste is then processed or tested in a support disk (as a battery).

4.3.3 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 5, based on discussions with the researcher, observations of the process, analysis of the starting materials (carbon/acetylene black and lithium compounds), and review of other reference documents. A summary of the analyses performed on the carbon (acetylene) black and lithium compounds is provided in Appendix B.

Lithium metal is stored in unopened containers on shelves with other chemicals or in an inert atmosphere glove box. Containers of lithium are opened only within the glove box and lithium is removed from the glove box only in sealed disposal containers or in electrochemical cells. Lithium is disposed of by packing it (while still inside the glove box) in mineral oil in cans, jars, or bottles with tight-fitting lids made of plastic, metal, or glass. These waste containers are then disposed of by routine LBNL procedures. Carbon (acetylene) black is stored in containers or plastic bags on shelves in Lab 299 and in containers inside the glove box.

Because the UNP is handled in glove boxes, there appears to be little opportunity for any worker exposure. The loss of UNP from the glove box into the exhaust air is also unlikely, but should be quantified through further study. Although the practice of exhausting the glove box vacuum system into the building exhaust ventilation system is good, and greatly reduces the potential for occupational exposures, there is little documented ESH information on the movement of nanoparticles from the glove box environment through the vacuum system, and subsequent release into the ventilation system and the outside environment.

Table 5 Specific Risk Attributes Associated with Vincent Battaglia's UNP Research

Risk Attribute	Bldg 70 Labs 295/297/299 Carbon (Acetylene) Black	Bldg 70 Labs 295/297/299 Lithium Titanate	Bldg 70 Labs 295/297/299 Lithium Iron Phosphate	Bldg 70 Labs 295/297/299 Silicon
Particle size	Primary particles ~30–40 nm; agglomerated structures on the order of micrometers	Individual particles ranged from 50 nm to >5 µm; agglomerates on the order of micrometers	Particles average >100 nm, ranging from ~50 nm to 500 nm; rod-shaped particles measured ~100–200 nm in diameter; agglomerates on the order of micrometers	Primary particles ~10–50 nm; agglomerated structures on the order of micrometers
Particle morphology	Aciniform structures comprising rounded and irregularly shaped particles.	Irregular shaped agglomerated particles	Rounded and rod- shaped particles; trace amount of angular particles	Aciniform structures comprising rounded particles, often spherical
Elemental chemistry	SEM/EDS: C	SEM/EDS: Ti, O; trace Si; Zr ICP-OES: Major Ti, Li, Si; trace Zr, Na, P, Fe, Al, Ni	SEM/EDS: Fe, P, O ICP-OES: Major Fe, P, L; trace Mn, Si, S, Al, Cr, K, Zn, Na	SEM/EDS: Si, O ICP-OES: Major Si; trace Na, P, B
Solubility (water)	Insoluble	Insoluble	Insoluble	Insoluble
Toxicity	Medium	Unknown	Unknown	Unknown
Amount of material used	< 10 g	< 10 g	< 10 g	< 10 g
Dustiness/airborne potential	Medium	High	High	Moderate
Number of people doing the work	3	1	2	1–3
Duration of the operation	5 min	10 min	10 min	< 10 min
Frequency of the operation	2 x/week	1 x/year	2 x/year	1 x/month

4.3.4 Process Overview: Nanosilicon and Other Nanopowders

The Battaglia group also works in fume hoods in the Battery Fabrication Laboratory with nano-size powders such as silicon, carbon (acetylene) black, and lithium compounds. Photos illustrating the process as demonstrated in the fume hood by Honghe Zheng in Lab 299 are provided in Appendix F.

Xiangyun Song simulated a process involving dry silicon powder, approximately 30 nanometers in diameter, in a fume hood in Building 70, Lab 299. The silicon powder is weighed and placed on a copper substrate using a metal spatula. Photos illustrating this process are provided in Appendix G.

Results

Another process was demonstrated in the fume hood in Building 70, Lab 299, in which dry powder, such as carbon black, silicon, or lithium metal such as $\text{Li}_4\text{Ti}_5\text{O}_{12}$, is funneled into a glass volumetric flask for analysis using a Brunauer, Emmett, and Teller (BET) instrument in Building 70, Lab 206. Photos illustrating the process are provided in Appendix H.

4.3.5 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 5, based on discussions with the researcher, observations of the process, analysis of the starting materials (carbon [acetylene] black, lithium compounds, and silicon), and review of reference documents. A summary of the analyses performed on carbon (acetylene) black, lithium compound, and silicon samples are provided in Appendix B.

Nanosilicon powder is stored inside a glove box (see the previous discussion of glove box use by the Battaglia Group). Work with the powder is performed in a functional fume hood. Appropriate work practices are being followed, so no significant occupational exposure would be anticipated. The fume hood is not HEPA-filtered, so a small amount of nanomaterial could be released to the environment during transfer of the materials. Any loss would be expected to be small based on duration and frequency of this activity (less than 10 minutes and occurring once a month). The significance of environmental release through the exhaust air will be studied further in Phase III of this program.

4.4 Vincent Battaglia: Building 70, Lab 206

4.4.1 Battery Materials Program

During the inspection of the Battery Fabrication Laboratory, researchers explained that material that is prepared in a fume hood in Building 70, Lab 299, and placed in a volumetric flask, is subsequently analyzed in Lab 206. To learn more about this process, the investigators visited Lab 206.

4.4.2 Process Overview: BET Analysis

In Lab 206, the material contained in the volumetric flask is analyzed for surface area and porosity using the gas adsorption technique in a Micromeritics BET model Tristar 3000 (V6.03) instrument.. The analysis consists of placing the volumetric flask in the analysis position in the BET (particulate matter is not removed from the flask). Photos illustrating the process are provided in Appendix I.

4.4.3 Discussion of Risk

Evaluation of the process as demonstrated by Xiangyun Song indicates that there is no significant potential for exposure or environmental release of UNP (barring accidental release through breakage of the volumetric flask). The Micromeritics BET instrumentation is used infrequently—just once or twice a year. Disposal of the specimen is accomplished through the LBNL hazardous waste disposal system which requires communication of the presence or potential presence of UNP.

4.5 Gao Liu: Building 70, Lab 226

During evaluation of UNP in Building 70, it was learned that research on single-wall carbon nanotubes (SWCNT) was previously performed under the direction of Gao Liu in Lab 226. To evaluate this further, the laboratory was inspected. No work related to nanoparticles is currently being performed in this laboratory. A small container of SWCNT from the previous research is being stored in a glass desiccator. Photos of the SWCNT container in the desiccator are provided in Appendix J.

4.5.1 Discussion of Risk

Because the SWCNT is being stored in a sealed container in an enclosed desiccator, occupational exposures or airborne environmental releases of UNP are not expected. No samples of starting materials were collected. This laboratory will not be evaluated further in subsequent phases of the study.

4.6 Robert Kostecki: Building 70, Lab 108

4.6.1 Battery Applications Program

Robert Kostecki's research is directed toward the optimization of the structure of carbon in lithium-ion composite electrodes intended for use in battery applications. The nanomaterials used in this research are lithium metal phosphate and carbon black. Currently the metal in the lithium compound is iron; manganese and other metals may be used in future research.

4.6.2 Process Overview: Lithium Compounds and Carbon Black

Although this work was not observed, Robert Kostecki works with lithium compounds and carbon black additives to make electrodes in exactly the same way that Vince Battaglia's group does, and often does this work in the Battery Fabrication Laboratory.

4.6.3 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 6, based on discussions with researchers, observations of the process, analysis of the starting material (lithium iron phosphate), and review of reference documents. The attributes assigned to carbon (acetylene) black in Table 6 are the same as those for Vince Battaglia's group (from Table 5). A summary of the analyses performed on the lithium iron phosphate sample is provided in Appendix B.

Since the UNP is handled in a glove box, there should be little opportunity for any worker exposure. The operations are conducted by 1–3 researchers working for less than 30 minutes, 1–5 times a week. The loss of UNP from the glove box into the exhaust air is also unlikely, but should be quantified through further study. Although the practice of exhausting the glove box vacuum system into the building exhaust ventilation system is good, and greatly reduces the potential for occupational exposures, further study is suggested. There is little documented ESH information on the movement of nanoparticles from the glove box environment through the vacuum system, and subsequent release into the ventilation system and the outside environment.

4.6.4 Graphene Research Program

Under Robert Kostecki's direction, Elad Pollak performs research related to graphene at this location. Graphene is the two-dimensional crystalline form of carbon whose extraordinary electron mobility and other unique features hold great promise for nanoscale electronics and photonics. However, as noted previously in this report (see the discussion of Building 62, Lab 342), it is not obvious whether graphene should be considered a nanoparticle. In Elad's research, only one dimension would have the potential to be in the nano-size range and the other dimensions would be on the order of millimeters in size (not respirable).

Table 6 Specific Risk Attributes Associated with Robert Kostecki's UNP Research

Risk Attribute	Building 70 Labs 295/297/299 Carbon (Acetylene) Black	Building 70 Labs 295/297/299 Lithium Iron Phosphate	Building 70 Lab 108 Graphene
Particle size	Primary particles ~30–40 nm; agglomerated structures on the order of micrometers	Majority of particles were > 100 nm. Individual particles ranged from ~50 nm to 500 nm; elongated particles often measured 100–200 nm in diameter and > 500 nm in length; agglomerated structures on the order of micrometers.	Individual platelets were agglomerated and could not be distinguished, size information was difficult to determine; individual platelets were on the order of 200 nm
Particle morphology	Aciniform structures comprising rounded and irregularly shaped particles	Elongated particles that were usually agglomerated	Slender platelets that appear to be agglomerated; unable to distinguish individual sheets
Elemental chemistry	SEM/EDS: C	SEM/EDS: Fe, P, O; Trace: Salt and carbon particles; ICP-OES: Major: Fe, P, Li; trace: Si, Mn, S, Al, Ca, Cr, Na, Zn	SEM/EDS: C
Solubility (water)	Insoluble	Insoluble	Insoluble
Toxicity ¹	Medium	Unknown	Low
Amount of material used	< 10 g	< 10 g	< 10 ng
Dustiness/airborne potential	Moderate	High	Moderate
Number of people doing the work	3	1–3	1
Duration of the operation	5 min	< 30 min	5 min
Frequency of the operation	2 x/week	1–5 x/week	2 x/month

4.6.5 Process Overview: Graphene

Elad's activities with graphene involve making one-atom thick carbon layers using adhesive tape. The process is described in detail at a website sponsored by the journal *Scientific American* (Minkel 2008). Photos showing the materials used in this process are provided in Appendix K.

4.6.6 Discussion of Risk

Specific risk attributes for this research process are provided in Table 6, based on discussions with the researcher, observations of the process, analysis of the starting material (graphene), and review of reference documents. A summary of the analyses performed on the graphene sample are provided in Appendix B.

Results

The “thinning” process used to make one-atom thick carbon layers does not appear to have the potential to be of concern from a personal exposure or environmental release perspective (especially since the graphene may not be considered a nanoparticle). In Phase III, the process will be sampled in an effort to determine whether graphene breaks apart into a nanoparticle (two dimensions less than 100 nm).

4.7 Samuel Mao: Building 70, Lab 163

4.7.1 Fuel Cell Program

Research conducted in this laboratory is directed by Samuel Mao and focuses on development of cost-effective materials for fuel cells. In this research, nanomaterials may be inadvertently created when pulsed lasers are used to vaporize a solid target (e.g., a solid TiO₂ target).

4.7.2 Process Overview: Vapor Condensate

During this process, part of the vapor will condense and some will solidify into nanoparticles (approximately 100 nm in size), which are then deposited onto a solid substrate. Depending on the experimental parameters, the laser-produced nanoparticles may coalesce and form a dense film on the substrate (e.g., TiO₂ thin film on silicon wafer). The final product is a continuous metallic layer that does not contain UNP. The entire process is performed in a sealed vacuum chamber operating at 10⁻⁷ T.

Zhixun Ma provided an overview of the process. Photos of the instrumentation used in this laboratory are provided in Appendix L.

4.7.3 Discussion of Risk

This process is fully enclosed in a vacuum chamber and presents no personnel exposure or environmental release of UNP during synthesis. Since the deposition process involves agglomeration of fumes (similar to application of a weld), it is not expected that UNP would be present in the laboratory air or in the exhaust air stream. No samples of starting materials were collected related to this process, so specific risk attributes for this work will not be available until Phase III sampling.

Contact with the deposited materials could occur during cleaning of the vacuum chamber. Cleaning of the vacuum chamber after using cadmium deposition (a worst-case scenario) has been evaluated by the LBNL Industrial Hygiene Group. Air monitoring for cadmium indicated very low exposures to the LBNL staff during cleaning. While personnel exposure to UNP and release to the ventilation exhaust is unlikely, air sampling for UNP within the vacuum duct exhaust will be performed in Phase III to provide verification and documentation.

4.8 Rick Russo: Building 70, Lab 157

4.8.1 Laser Ablation Program

Research conducted in this laboratory is directed by Rick Russo and performed by Xianglei Mao. The research focuses on fundamental studies related to laser ablation. In this research, nanomaterials may be inadvertently created during laser ablation.

4.8.2 Process Overview: Vapor Condensate

Lasers used for these studies include a nanosecond Nd:YAG and a femtosecond Ti:Sapphire. During laser ablation, target materials go through a phase change where molecules, atoms, and ions are free in a plasma, but they are almost instantaneously agglomerated into particles that are larger than nanoparticles. Photos of the instrumentation used in this laboratory are provided in Appendix M.

4.8.3 Discussion of Risk

Because this research is performed in an enclosed vacuum chamber and a negative-pressure enclosure, occupational exposures or airborne environmental release of UNP are unlikely. Cleaning and maintenance of equipment may present an exposure to the materials deposited within the apparatus. While personnel exposure to UNP and release to the ventilation exhaust is unlikely, air sampling for UNP within the vacuum duct exhaust will be performed in Phase III to provide verification and documentation.

4.8.4 Student Research Project

During the investigation of Lab 157, it was determined that Matt Lucas works with nanomaterials in this laboratory. While the interviewers understood that Matt is studying colloidal nano-size gold, subsequent discussions with Matt and Xianglei Mao indicate that iron disulfide, not gold, is being studied in this laboratory. Photos showing Matt discussing the process are provided in Appendix N.

4.8.5 Process Overview: Student Research with Nanomaterials

Matt described the process which consisted of placing an aliquot of the nanomaterial in solution in a sample cuvette inside the lab hood. He noted that he sometimes uses compressed air to dry the suspension.

4.8.6 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 7, based on discussions with the researcher, analysis of the starting materials (iron disulfide), and review of reference documents. A summary of the analyses performed on the iron disulfide sample is provided in Appendix B.

Table 7 Specific Risk Attributes Associated with Rick Russo's UNP Research

Risk Attribute	Building 70 Lab 157 Iron Disulfide
Particle size	Droplets measured ~20 to 80 nm; agglomerated structures on the order of micrometers
Particle morphology	Majority of the particles by number consisted of Fe-S in the form of a droplet; agglomerated Fe-S particles on the order of micrometers also present
Elemental chemistry	SEM/EDS: Major: Fe-S; Minor: Ca-Fe-S; Trace: Si
Solubility (water)	Insoluble
Toxicity	Unknown
Amount of material used	< 100g
Dustiness/airborne potential	High
Number of people doing the work	1
Duration of the operation	< 10 min
Frequency of the operation	1–5 x/week

This process is performed within an approved, functional hood, thus no significant occupational exposure would be anticipated. However, UNP may be released to the ambient environment through the ventilation exhaust (the hood is not filtered). Any loss of UNP would be expected to be small based on duration and frequency of this activity (less than a 10-minute process that occurs 1–5 times a week). The significance of the presence of iron disulfide in the exhaust air will be studied further in Phase III of this pilot study.

4.9 Don Lucas: Building 70, Labs 291/293

4.9.1 Detection of Toxic Species

Research directed by Don Lucas is being performed to develop better methods to detect toxic species (e.g., mercury) using optical sensors. This research employs nano-size gold rods and spheres (~12 nm size).

4.9.2 Process Overview: Gold Rods/Spheres

An overview of the process was provided by Jay James, who discussed how gold rods and spheres are suspended in an aqueous solution with a surfactant and placed on a substrate for testing and analysis. Photos taken during Jay's explanation of the process are provided in Appendix O.

4.9.3 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 8, based on discussions with the researcher, observations of the process, analysis of the starting materials (nanogold samples), and review of reference documents. A summary of the analyses performed on the nanogold samples are provided in Appendix B.

The lab hood is functional, and appropriate work practices are being followed, so no significant occupation exposure would be anticipated. The potential for release of the nanoparticles to the air and subsequent exposure to the environment is low, especially considering that the researcher is working with an aqueous solution, the duration of the activity is less than 10 minutes, and it occurs 1–5 times a week. The fume hood will be subject to further monitoring in Phase III to document potential UNP release.

4.9.4 Combustion Diagnosis

Research conducted in the Combustion Diagnostic Laboratory is directed by Don Lucas and focuses on small-scale experiments using lasers to detect and measure species produced during combustion. With respect to nanoparticles, the focus of this research is carbon soot.

4.9.5 Process Overview: Carbon Soot

An enclosed apparatus is used to synthesize soot, and some percentage of the particles may be in the nano-size range. The soot generated in this research is subjected to several forms of analysis including use of the scanning mobility particle sizer (SMPS) nanoparticle analyzer and BET analysis. Because of the dynamics of soot generation, synthesized particles agglomerate and take on characteristics of larger airborne particles (not nanoparticles). Photos of the apparatus used to generate the soot are provided in Appendix P.

Table 8 Specific Risk Attributes Associated with Don Lucas's UNP Research

Risk Attribute	Building 70 Labs 291/293 Gold Nanorods	Building 70 Labs 291/293 Gold Nanospheres	Building 70 Labs 291/293 Oxidized Soot
Particle size	Rod-shaped particles ~20 nanometers (nm) in diameter and ~50 nm in length; rounded and spherical particles were ~40–50 nm in diameter	Sizes ranged from ~3 to 125 nm	Primary particles ~20–40 nm; agglomerated structures on the order of micrometers
Particle morphology	Primarily rod-shaped particles; rounded and spherical particles; observed in clusters	Irregularly shaped and angular particles were predominant in the larger size fraction; particles <~50 nm were spherical; a trace of rod-shaped particles were detected	Aciniform structures comprising rounded and irregularly shaped particles
Elemental chemistry	SEM/EDS: Au; Si residue	SEM/EDS: Au; Si residue	SEM/EDS: C
Solubility (water)	Insoluble	Insoluble	Insoluble
Toxicity	High	High	Medium
Amount of material used	< 10 mg	< 10 mg	< 1 g
Dustiness/airborne potential	Low	Low	Low
Number of people doing the work	1–3	1–3	1–3
Duration of the operation	< 10 min	< 10 min	< 60 min
Frequency of the operation	1–5 x/week	1–5 x/week	1 x/week

4.9.6 Discussion of Risk

Specific risk attributes identified for this research process are provided in Table 8, based on discussions with the researcher, observations of the process, analysis of the starting material (oxidized soot), and review of reference documents. A summary of the analyses performed on the oxidized soot sample is provided in Appendix B.

Given the nature of the research design, occupational exposures to nanoparticles would not be significant. Further study will be conducted in Phase III to verify worker safety and the potential for release of soot into the exhaust ventilation system.

4.10 Andre Anders: Building 70, Lab 274

4.10.1 Sputter Deposition Facility

During the investigation of laboratories in Building 70, discussion turned to a sputter deposition system to produce thin films of various material, used by Jonathan Slack in Andre Anders' lab (274). To learn more about the instrumentation and its use, an impromptu phone interview was arranged with Jonathan. He stated during the interview that in the past, NiO- and Li-bearing compounds were sputtered with this instrument. Currently, the facility is used daily for deposition of Ag, Au, Sb, Sn, Cu, Mn, Mg, Ti, Al, V, Si, and Zr oxides.

4.10.2 Process Overview: Metal Sputtering

Because the work is conducted in a sealed vacuum chamber, any particles released would occur only during cleaning, and cleaning occurs only about once every year or two. Chamber cleaning is planned and documented in a site safety procedure. Air monitoring during chamber cleaning has shown no significant personnel exposure. Photos of the instrumentation are provided in the Appendix Q.

4.10.3 Discussion of Risk

Because this research is performed in an enclosed vacuum chamber, occupational exposures or airborne environmental releases of UNP are unlikely, especially considering that the instrumentation is infrequently cleaned. No samples of starting materials were collected. This laboratory will not be evaluated further in subsequent phases of the study.

5.0

Conclusions

In Phase I of the pilot study, work with nanomaterials conducted by the following nine principal investigators in EETD was reviewed.

1. John Kerr, Building 62, Lab 246
2. Thomas Richardson, Building 62, Lab 342
3. Vincent Battaglia, Building 70, Labs 295/297/299 and 206
4. Gao Liu, Building 70, Lab 226
5. Robert Kostecki, Building 70, Lab 108
6. Samuel Mao, Building 70, Lab 163
7. Rick Russo, Building 70, Lab 157
8. Don Lucas, Building 70, Labs 291/293
9. Andre Anders, Building 70, Lab 274

After interviews and demonstrations, work with UNP done under two principal investigators, Gao Liu and Andre Anders, was determined to be unlikely sources of occupational exposure or environmental releases of UNP by any release route. This work will not be considered in subsequent phases of the pilot study.

Samples of the researchers' starting materials were analyzed and the following hazard and exposure attributes were determined.

1. particle size
2. particle morphology
3. elemental chemistry
4. solubility
5. toxicities of starting materials
6. amount of material used
7. dustiness (or potential for the material to become airborne based on the task/process)
8. number of people doing the work
9. duration of the operation
10. frequency of the operation

This information will be used in Phase II of the pilot study to identify health/environmental hazards and exposure/release potential, and to develop preliminary control bands.

6.0

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Appendix A

Samples of Starting Materials Received for Analyses

Date Received	Principal Investigator	Building	Laboratory	Sample Description	Medium
5-Aug-09	John Kerr	62	246	Fumed silica	dry
5-Aug-09	John Kerr	62	246	Carbon (Acetylene) Black	dry
27-Jul-09	Thomas Richardson	62	342/344	nanoAu	in water
27-Jul-09	Thomas Richardson	62	342/344	nanoAg	in water
27-Jul-09	Thomas Richardson	62	342/344	graphene	dry
27-Jul-09	Thomas Richardson	62	342/344	Acetylene black	dry
27-Jul-09	Thomas Richardson	62	342/344	Carbon black	dry
5-Aug-09	Vince Battaglia	70	295/299	Carbon (Acetylene) Black	dry
5-Aug-09	Vince Battaglia	70	295/299	Lithium titanate powder	dry
5-Aug-09	Vince Battaglia	70	295/299	Lithium iron phosphate powder	dry
5-Aug-09	Vince Battaglia	70	295/299	Silicon powder	dry
7-Aug-09	Robert Kostecki	70	108	Graphene	on Scotch Tape
7-Aug-09	Robert Kostecki	70	108	Lithium iron phosphate	dry
7-Aug-09	Rick Russo	70	157	Iron Disulfide	dry
5-Aug-09	Don Lucas	70	291/293	Au nanorods	aqueous
5-Aug-09	Don Lucas	70	291/293	Au nanospheres	aqueous
5-Aug-09	Don Lucas	70	291/293	Oxidized Soot	dry

Appendix B

Results of Starting Materials Analyses

- B1: John Kerr's Sample Analyses
- B2: Thomas Richardson's Sample Analyses
- B3: Vincent Battaglia's Sample Analyses
- B4: Robert Kostecki's Sample Analyses
- B5: Rick Russo's Sample Analyses
- B6: Don Lucas's Sample Analyses



September 18, 2009

Ms. Linnea Wahl
Pilot Study Project Manager
Ernest Orlando Lawrence Berkeley National Laboratory
Berkeley, CA 94720

Re: Electron Microscopy Results of Potential UNP
PI: John Kerr: Building 62, Lab 246

Dear Linnea:

This report summarizes the electron microscopy (EM) results for the analysis of the samples collected from Building 62, Lab 246 as part of the Phase I pilot study related to potential unbound engineered nanoparticle (UNP) releases. Research conducted in this laboratory is directed by John Kerr and focuses on battery materials. Nanomaterials used in the process include fumed silica and acetylene black (carbon nanoparticles). Information related to the samples is listed in Table 1. The EM analysis was performed to provide information on particle size, morphological characteristics, and elemental composition associated with each material.

Table 1: Sample Information

Client Sample ID	RJLG Sample #	Sample Received Date	Sample Analysis Date	Analyst
Fumed Silica	10066040	August 5, 2009	September 2, 2009	Kristin Bunker
Acetylene Black	10066039	August 5, 2009	September 2, 2009	Kristin Bunker

The samples were characterized using a Hitachi S-5500 high resolution field emission scanning electron microscope (HR-FESEM) with scanning transmission electron microscopy (STEM) capabilities. The analysis was conducted primarily in the secondary and bright field transmission electron modes at magnifications up to 1,000,000x. Compositional information was obtained through collection and processing of characteristic X-rays using a Bruker energy dispersive X-ray spectroscopy (EDS) system incorporating a 30 mm² silicon drift detector (SDD).

The fumed silica sample was analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES). A Varian 730ES ICP-OES was used to detect and quantify the elements in the material including trace metals by measuring light emitted at wavelengths characteristic of individual elements. The intensity of this emission is indicative of the concentration of the element within the sample.

The samples were prepared for ICP-OES using acid digestion techniques. The limited sample size and the unique composition of the samples did not allow for the preparation/digestion of any matrix duplicate (MD), matrix spike (MS) and laboratory control standard (LCS) quality control samples. A laboratory reagent blank (LRB) along with a fortified laboratory reagent blank (LFB) were prepared and analyzed instead. Elevated silicon (1.864 mg/L) in the LRB results was due to etching of the glass in the ICP-OES system by the hydrofluoric acid (HF) used to digest the samples. Elevated phosphorus (0.055 mg/L) in the LRB results was due to the sample container used to perform the digestion. All of the recoveries reported in the LFB results were within the acceptable laboratory limits ($\pm 15\%$), with the exception of silicon (for the same reasons as described above for the LRB) and calcium (which tends to form insoluble CaF_2 in the presence of HF). A detailed description of the sample preparation is described along with the results of the analysis in the table attached to this report.

The table below (Table 2) provides a concise summary of the EM analysis results. The sections following the table provide a more detailed summary of the EM results for the fumed silica and the acetylene black (carbon nanoparticles).

Table 2: Sample Attributes

	Particle Size	Morphology	Chemistry
Fumed Silica	Primary particles ~20-30 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of rounded particles	SEM/EDS: Si, O and trace amounts of Cl, K, Na, Ca, S, F ICP-OES: Major element was Si and trace amounts of Na, P, B
Acetylene Black	Primary particles ~30-40 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of rounded and irregularly shaped particles	SEM/EDS: C

Fumed Silica Sample

The fumed silica dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The EM analysis revealed that the primary particles were 20-30 nanometers (nm) in diameter with a rounded shape and a smooth surface texture. The particles were observed as agglomerates usually in the micrometer size range. The EDS analysis of the material revealed that the particles were composed of silicon (Si) and oxygen (O) with traces amounts of chlorine (Cl), potassium (K), sodium (Na), calcium (Ca), sulfur (S), and fluorine (F). EM micrographs and elemental spectra of the fumed silica agglomerates are presented in Figure 1 and Figure 2. Figure 3 illustrates the primary particles at an increased magnification.

The fumed silica sample was also analyzed by ICP-OES. The sample was comprised of Si with trace amounts of Na, phosphorus (P), and boron (B). The results can be found at the end of the report.

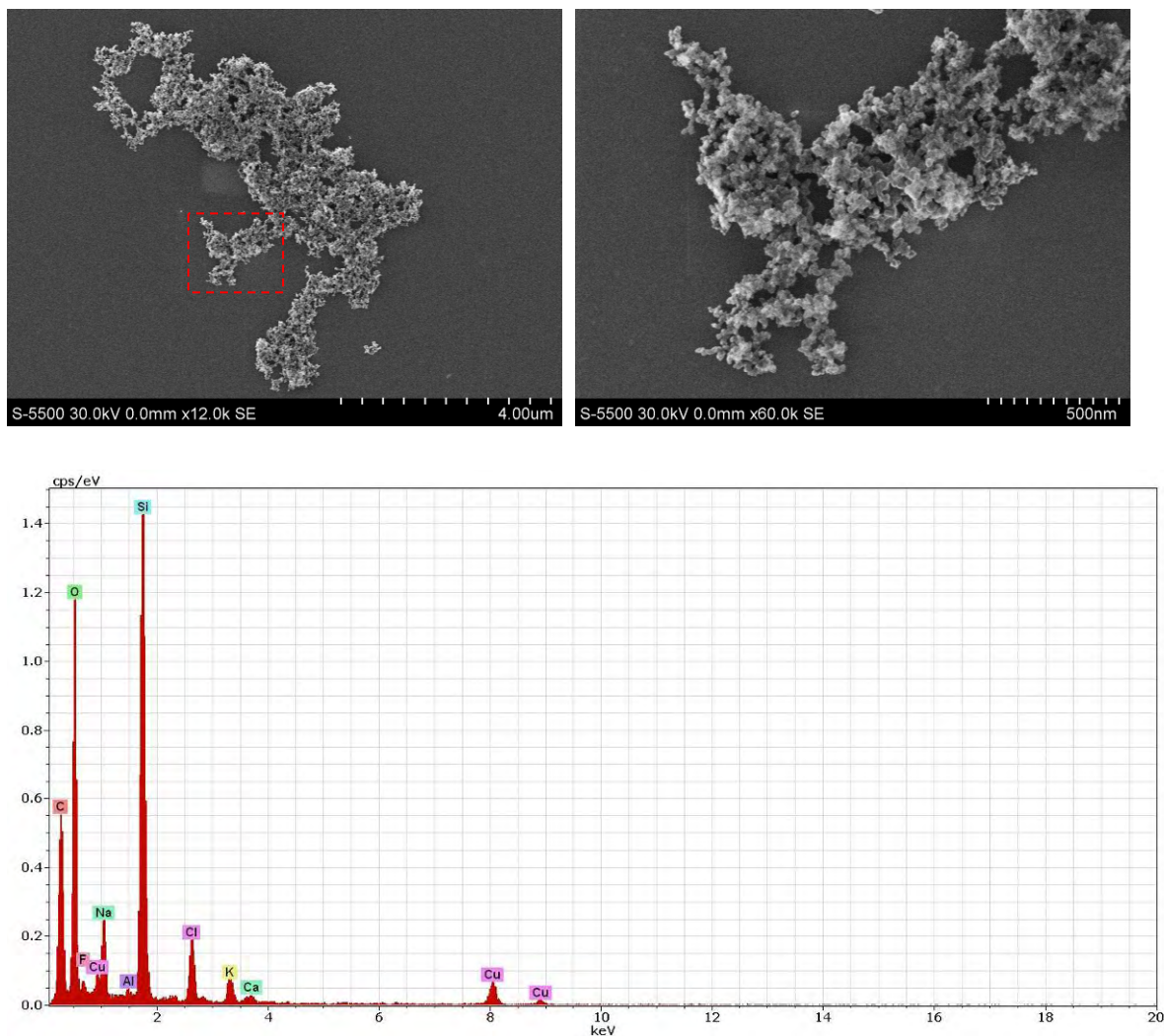


Figure 1. Secondary electron images along with an elemental spectrum of the fumed silica nanoparticles; (copper (Cu) X-rays are fluoresced from the TEM grid, carbon (C) from the substrate material, and aluminum (Al) from the sample holder).

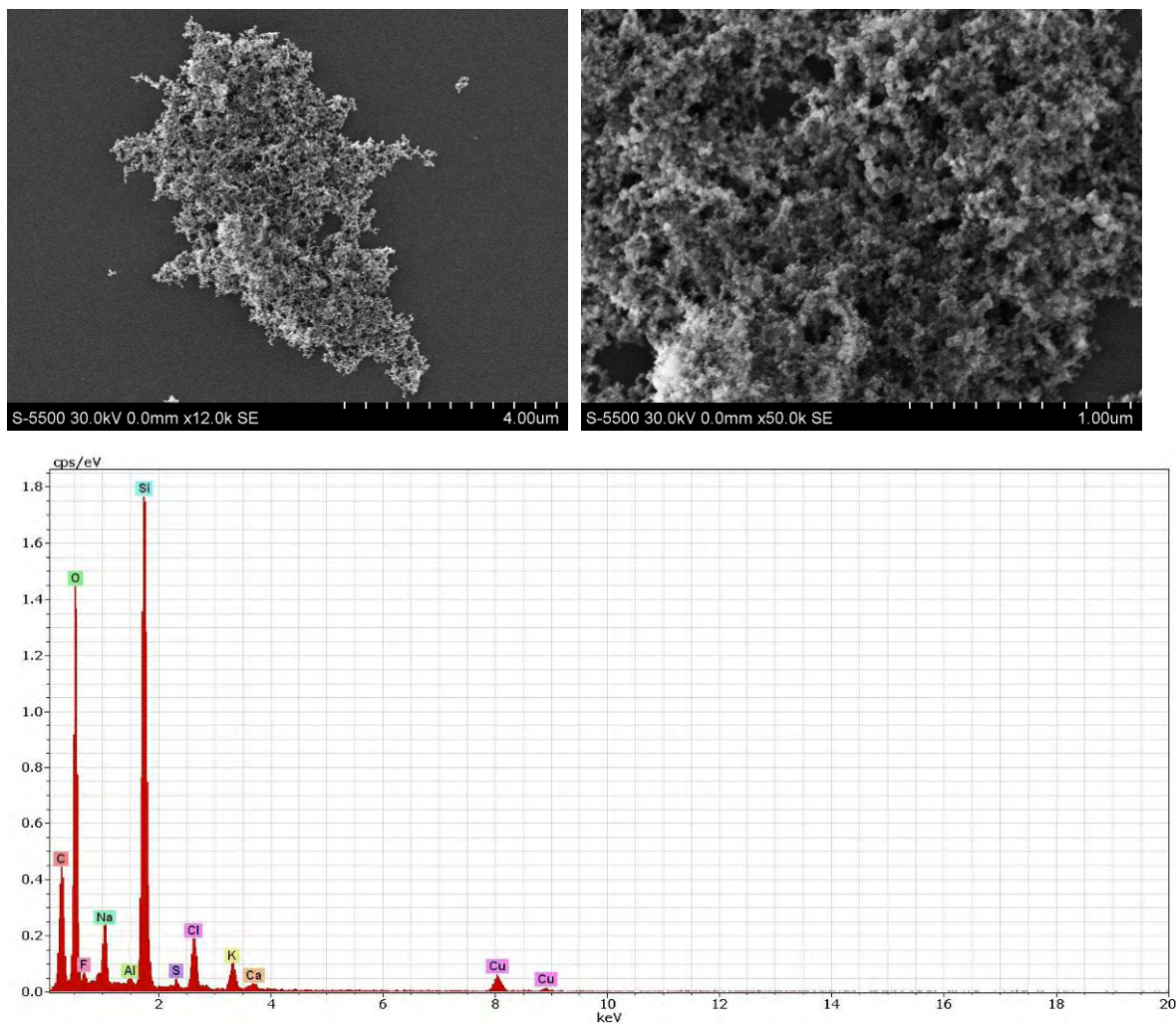


Figure 2. Secondary electron images along with an elemental spectrum of the fumed silica nanoparticles.

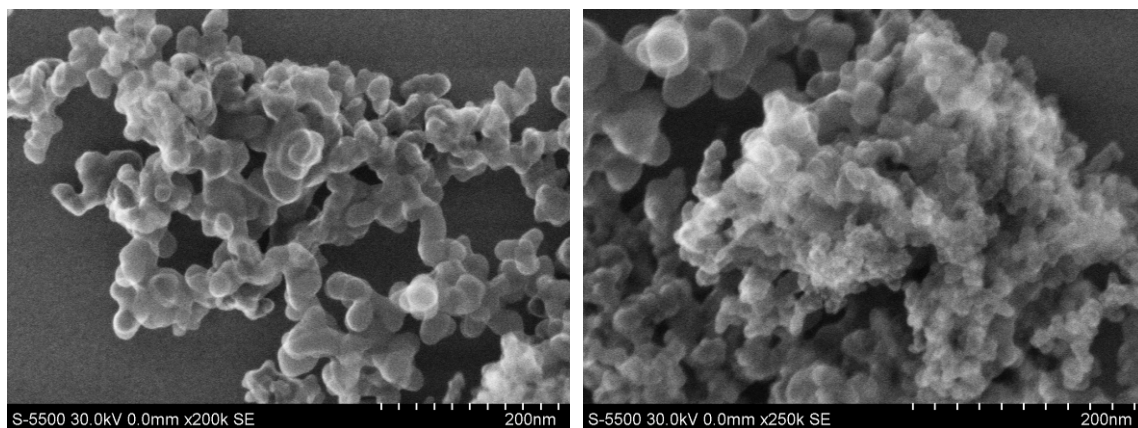


Figure 3. Secondary electron images of the fumed silica nanoparticles.

Acetylene Black (Carbon Nanoparticles)

The acetylene black dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A lacy Formvar/carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The sample was comprised of aciniform structures consisting of agglomerated irregular and rounded primary particles that averaged approximately 30-40 nm in diameter. The particles were observed as agglomerates usually in the micrometer size range. The EDS analysis of the material confirmed that the particles were composed of carbon. Primary particles were well defined with a smooth particle perimeter per ASTM 6602- 03b¹. EM micrographs and an elemental spectrum of the aciniform structures are presented in Figure 4. Figure 5 illustrates the primary particles at an increased magnification. The microstructure of the acetylene black is shown in Figure 6.

¹ ASTM International Designation: D 6602 – 03b; Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both¹; ASTM Committee D24; Published January 2004.

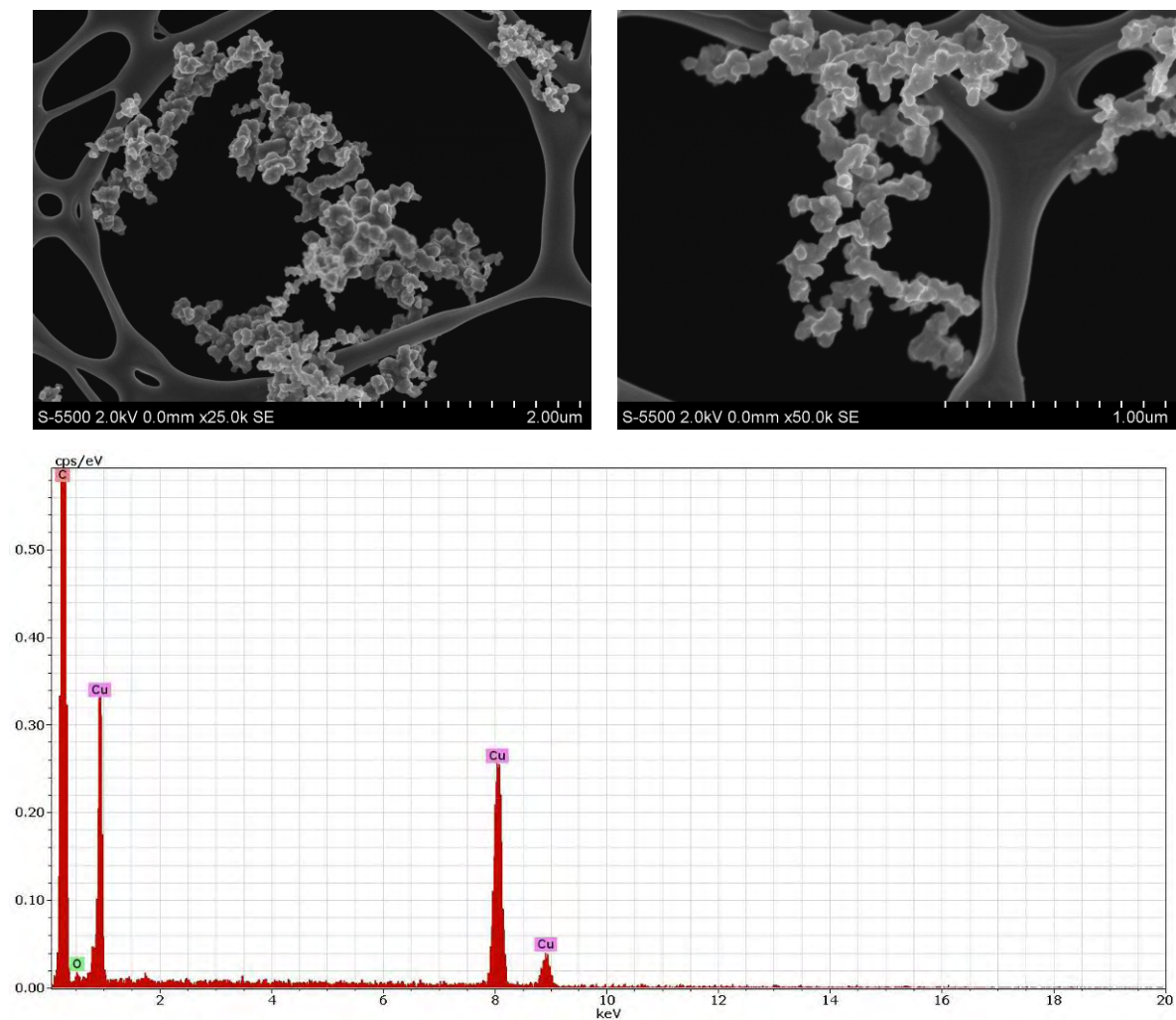


Figure 4. Secondary electron images along with an elemental spectrum of the acetylene black.

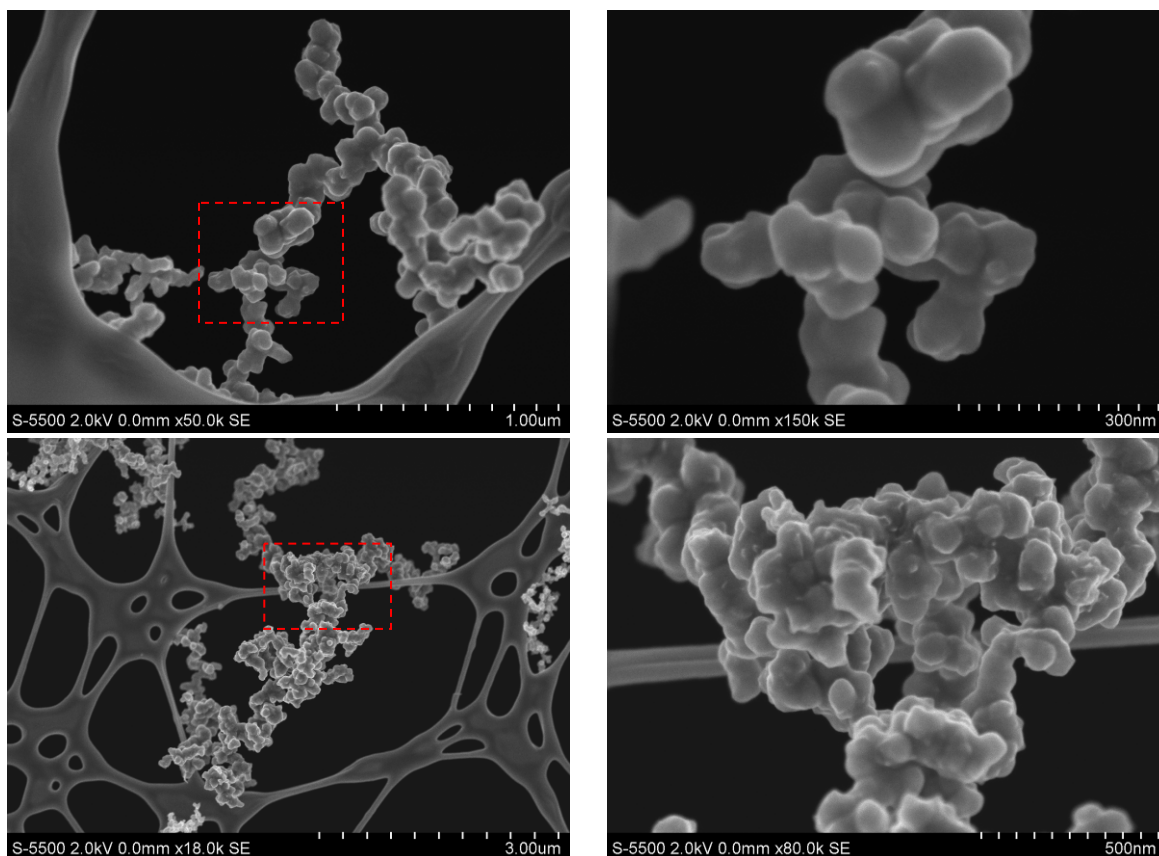


Figure 5. Secondary electron images of acetylene black.

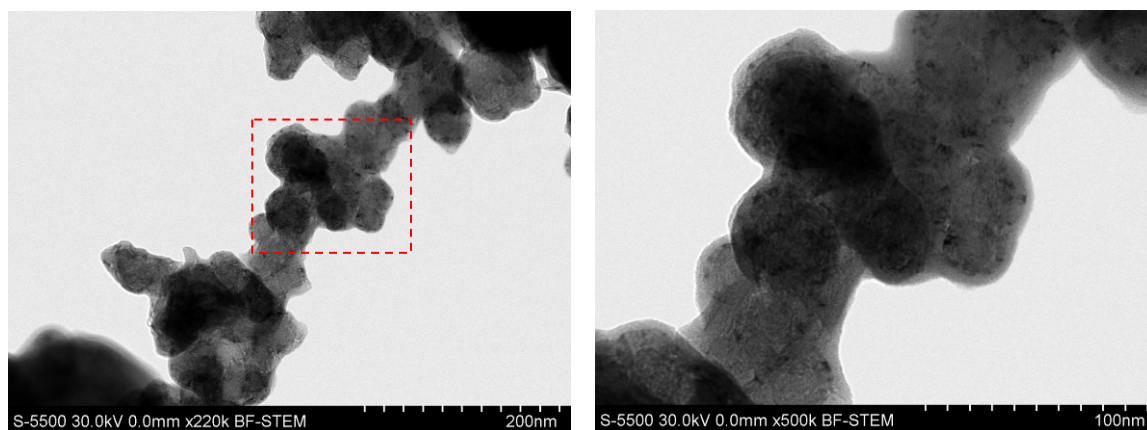


Figure 6. Bright field STEM images illustrating primary particle microstructure of acetylene black.

Ernst Orlando Lawrence Berkeley National Laboratory
RJ Lee Group Project No. GSG905991
September 18, 2009
Page 9 of 9

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Should you have any questions regarding this information, please do not hesitate to contact us.

Sincerely,

A handwritten signature in cursive script, reading "Kristin L. Bunker".

Kristin L. Bunker, Ph.D.
Senior Scientist

cc: G. Casuccio (RJLG)



LABORATORY REPORT

Lawrence Berkeley Lab
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MS 90J-01
Berkeley, CA 94720

Attn: Linnea Wahl
Phone: 510-486-7433
Fax: 510-486-6668
Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q	
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)			
B1 - 10	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Aluminum	< 0.0154	< 154	0.0154	154	09/08/2009	DS
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Antimony	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Arsenic	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Barium	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Beryllium	< 0.00247	< 24.7	0.00247	24.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Bismuth	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Boron	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Cadmium	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Calcium	< 0.0309	< 309	0.0309	309	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Chromium	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Cobalt	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Copper	< 0.00926	< 92.6	0.00926	92.6	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Iron	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Lead	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Lithium	< 0.00926	< 92.6	0.00926	92.6	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Magnesium	< 0.0309	< 309	0.0309	309	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Manganese	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Molybdenum	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Nickel	< 0.00309	< 30.9	0.00309	30.9	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Phosphorus	0.0230	230	0.00309	30.9	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Potassium	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Selenium	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Silicon	17.8	178000	0.617	6170	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Silver	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Sodium	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Sulfur	< 0.0309	< 309	0.0309	309	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Thallium	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Tin	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Titanium	< 0.0216	< 216	0.0216	216	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Vanadium	< 0.00309	< 30.9	0.00309	30.9	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Zinc	< 0.0154	< 154	0.0154	154	09/08/2009	
	Fumed Silica Nanopowder	10066040	N/A	John Kerr	Bldg. 62 Laboratory 246	Zirconium	< 0.00617	< 61.7	0.00617	61.7	09/08/2009	
											B	
											BS	



LABORATORY REPORT

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Berkeley, CA 94720

Attn: Linnea Wahl
Phone: 510-486-7433
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Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)		

Analyst Comments: ~20 mg of sample was placed in 50 mL centrifuge tube. 5 mL of nitric acid and 10 drops of hydrofluoric acid were added and they were digested for 1 hour at 100 °C. After that samples were cooled down, 3 mL of hydrochloric acid was added and samples were returned to hot block for another 30 minutes at 100 °C. Final volume was 25 mL. The digested samples were analyzed in a Varian 730ES ICP-OES (Inductive Coupled Plasma - Optical emission Spectroscopy).

Report Qualifiers (Q):

H = Holding times for preparation or analysis exceeded

P = NELAC[®] analyte certification pending

N = Analyte not NELAC[®] certified

*NELAC-National Environmental Laboratory Accreditation Conference

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.

This laboratory operates in accord with ISO 17025:2005 guidelines, and holds a limited scope of accreditation under AIHA Lab ID 100364, NY ELAP Lab Code 10884, EPA Lab Code PA00162, CA ELAP Certificate 1970, PA DEP Lab ID 02-00396, VA DCLS Lab ID 00297, and LA DEQ Agency Interest 94775. This report may not be used to claim product endorsement by any laboratory accrediting agency. The results contained in this report relate only to the items tested or to the sample(s) as received by the laboratory. Any reproduction of this document must be in full for the report to be valid.

Quality Control data is available upon request. Results have not been blank corrected unless otherwise noted. Samples were received in good condition unless otherwise noted.

E = Value above highest calibration standard

J = Value below lowest calibration standard but above MDL (Method Detection Limit)

L = LCS (Laboratory Control Standard)/SRM (Standard Reference Material) recovery

outside accepted recovery limits

B = Analyte detected in the associated Method Blank

S = Spike Recovery outside accepted limits

R = RPD (relative percent difference) outside accepted limits

D = RL (reporting limit verification) outside accepted limits



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

Lawrence Berkeley Lab
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Berkeley, CA 94720

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RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID		Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
B1 - 12	PA-LRB:PAPB2-00909-03124-6300/QA1	Aluminum	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Antimony	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Arsenic	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Barium	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Beryllium	< 0.0080	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Bismuth	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Boron	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Cadmium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Calcium	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Chromium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Cobalt	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Copper	< 0.030	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Iron	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Lead	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Lithium	< 0.030	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Magnesium	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Manganese	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Molybdenum	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Nickel	< 0.010	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Phosphorus	0.055	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Potassium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Selenium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Silicon	1.864	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Silver	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Sodium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Sulfur	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Thallium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Tin	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Titanium	< 0.070	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Vanadium	< 0.010	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Zinc	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Zirconium	< 0.020	---	---	09/08/2009	



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

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Berkeley, CA 94720

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Phone: 510-486-7433
Fax: 510-486-6668
Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID		Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
B1 - 13	PA-LFB:PAPB2-00909-03124-6300/QA2	Aluminum	0.93	1	93	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Antimony	0.92	1	92	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Arsenic	0.897	1	89.7	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Barium	0.913	1	91.3	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Beryllium	0.859	1	85.9	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Bismuth	0.914	1	91.4	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Boron	1.018	1	101.8	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Cadmium	0.868	1	86.8	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Calcium	0.832	1	83.2	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Chromium	0.925	1	92.5	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Cobalt	0.91	1	91	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Copper	0.951	1	95.1	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Iron	0.888	1	88.8	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Lead	0.893	1	89.3	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Lithium	0.863	1	86.3	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Magnesium	0.877	1	87.7	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Manganese	0.925	1	92.5	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Molybdenum	0.952	1	95.2	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Nickel	0.889	1	88.9	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Phosphorus	0.968	1	96.8	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Potassium	9.333	10	93.3	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Selenium	0.874	1	87.4	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Silicon	1.171	1	117.1	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Silver	0.889	1	88.9	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Sodium	0.936	1	93.6	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Sulfur	0.921	1	92.1	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Thallium	0.872	1	87.2	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Tin	0.856	1	85.6	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Titanium	0.952	1	95.2	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Vanadium	0.944	1	94.4	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Zinc	0.869	1	86.9	09/08/2009	
	PA-LFB:PAPB2-00909-03124-6300/QA2	Zirconium	0.948	1	94.8	09/08/2009	



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

Lawrence Berkeley Lab
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Berkeley, CA 94720

Attn: Linnea Wahl
Phone: 510-486-7433
Fax: 510-486-6668
Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID	Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
-----------------	---------	--------------------------------	----------------------------	-----------------	------------------	---

Analyst Comments:

Report Qualifiers (Q):
H = Holding times for preparation or analysis exceeded
P = NELAC® analyte certification pending
N = Analyte not NELAC® certified
NELAC-National Environmental Laboratory Accreditation Conference
These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.
This laboratory operates in accord with ISO 17025 guidelines, and holds limited scopes of accreditation under NY ELAP Lab Code 10884, EPA Lab Code PA00162, CA ELAP Certificate 1970, PA DEP Lab ID 02-00396, VA DCLS Lab ID 00297, and LA DEQ Agency Interest 94775.
This report may not be used to claim product endorsement by any laboratory accrediting agency. The results contained in this report relate only to the items tested or to the sample(s) as received by the laboratory. Any reproduction of this document must be in full for the report to be valid. Quality Control data is available upon request.

E = Value above highest calibration standard but below LDR (Linear Dynamic Range)
J = Value below lowest calibration standard but above MDL (Method Detection Limit)
L = LCS (Laboratory Control Standard)/SRM (Standard Reference Material) recovery outside accepted recovery limits
B = Analyte detected in the associated Method Blank
S = Spike Recovery outside accepted recovery limits
R = RPD (relative percent difference) outside accepted recovery limits



September 18, 2009

Ms. Linnea Wahl
Pilot Study Project Manager
Ernest Orlando Lawrence Berkeley National Laboratory
Berkeley, CA 94720

Re: Electron Microscopy Results of Potential UNP
PI: Thomas Richardson: Building 62, Lab 342/344

Dear Linnea:

This report summarizes the electron microscopy (EM) results for the analysis of the samples collected from Building 62, Lab 342 as part of the Phase I pilot study related to potential unbound engineered nanoparticle (UNP) releases. Research conducted in this laboratory is directed by Thomas Richardson and focuses on battery materials and graphene. Nanomaterials being utilized in these research areas include silver, gold, graphene, acetylene black, and carbon black. Information related to the samples is listed in Table 1. The EM analysis was performed to provide information on particle size, morphological characteristics, and elemental composition associated with each material.

Table 1: Sample Information

Client Sample ID	RJLG Sample #	Sample Received Date	Sample Analysis Date	Analyst
Nano Ag	10066010	July 27, 2009	August 26, 2009	Traci Lersch
Nano Au	10066011	July 27, 2009	August 27, 2009	Kristin Bunker
Graphene	10066012	July 27, 2009	August 28, 2009	Traci Lersch
Acetylene Black	10066013	July 27, 2009	August 31, 2009	Traci Lersch
Carbon Black	10066014	July 27, 2009	August 31, 2009	Traci Lersch

The samples were characterized using an Hitachi S-5500 high resolution field emission scanning electron microscope (HR-FESEM) with scanning transmission electron microscopy (STEM) capabilities. The analysis was conducted primarily in the secondary electron and bright field transmission electron modes at magnifications up to 1,000,000x. Compositional information was obtained

through collection and processing of characteristic X-rays using a Bruker energy dispersive X-ray spectroscopy (EDS) system incorporating a 30 mm² silicon drift detector (SDD).

Additional analysis was conducted on the gold nanoparticle material on the Hitachi HD-2300 dedicated scanning transmission electron microscope (STEM) at an accelerating voltage 200 kV. The Hitachi HD-2300 is equipped with a Thermo energy dispersive X-ray spectroscopy system (EDS) and a silicon lithium detector.

The table below (Table 2) provides a concise summary of the EM analysis results. The sections following the table provide a summary of the EM results for the silver, gold, graphene, acetylene black, and carbon black samples.

Table 2: Sample Attributes

	Particle Size	Morphology	Chemistry
Nano Ag	Rounded and angular averaged <100 nm, ranging from ~10 nm to 150 nm; Rod-shaped were 50-75 nm in diameter and >500 nm in length; Agglomerated structures on the order of micrometers	Rounded, angular, and rod-shaped particles; often agglomerated	SEM/EDS: Ag with Si residue
Nano Au	Average 3 nm in diameter, ranging from 1.4-3 nm	Rounded particles, often spherical; also observed in clusters	SEM/EDS: Au
Graphene	Size information was difficult to determine; individual platelets appear on the order of 200 nm	Agglomerated slender platelets; unable to distinguish individual sheets	SEM/EDS: C with trace of O and Si
Acetylene Black	Primary particles ~30-40 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of irregularly shaped primarily particles	SEM/EDS: C
Carbon Black	Primary particles ~50-75 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of agglomerated irregular, angular, and rounded particles	SEM/EDS: C

Silver Sample

An aqueous solution (~ 2 ml) containing silver nanoparticles was provided for EM analysis. Sample preparation involved dispersing the particles in the solution using ultrasonic agitation. A carbon substrate supported by a transmission electron microscope (TEM) grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The EM analysis revealed that the majority of the sample was comprised of individual rounded particles and angular particles usually measuring less than 100 nanometers (nm), ranging from approximately 10 nm to 150 nm in average diameter. This observation is consistent with the MSDS information that indicates an average particle size of 20-100 nm. The EDS analysis of the material confirmed that the particles were composed of silver (Ag). The EM micrographs and elemental spectrum presented in Figure 1 illustrate exemplar particles. Straight cylindrical particles (rod-shaped) with rounded ends measuring 50-75 nm in diameter and greater than 500 nm in length were also detected. Rounded, angular, and rod-shaped particles each had a smooth surface texture and were often agglomerated. The size of these agglomerated structures was variable measuring on the order of micrometers in some cases. Figure 2 presents example images of agglomerated structures. The image shown in Figure 2a provides size overlays for a number of individual particles associated with an agglomerate that measured greater than 1 micrometer (μm) in the longest dimension. The particles were measured using an image analysis program (i.e., analySIS¹). Figure 3 illustrates a residual material associated with the sample comprised of silicon (Si), sodium (Na), sulfur (S), chlorine (Cl), fluorine (F), and oxygen (O).

¹ analySIS®; Digital Solutions for Imaging and Microscopy Soft Imaging System; www.soft-imaging.net

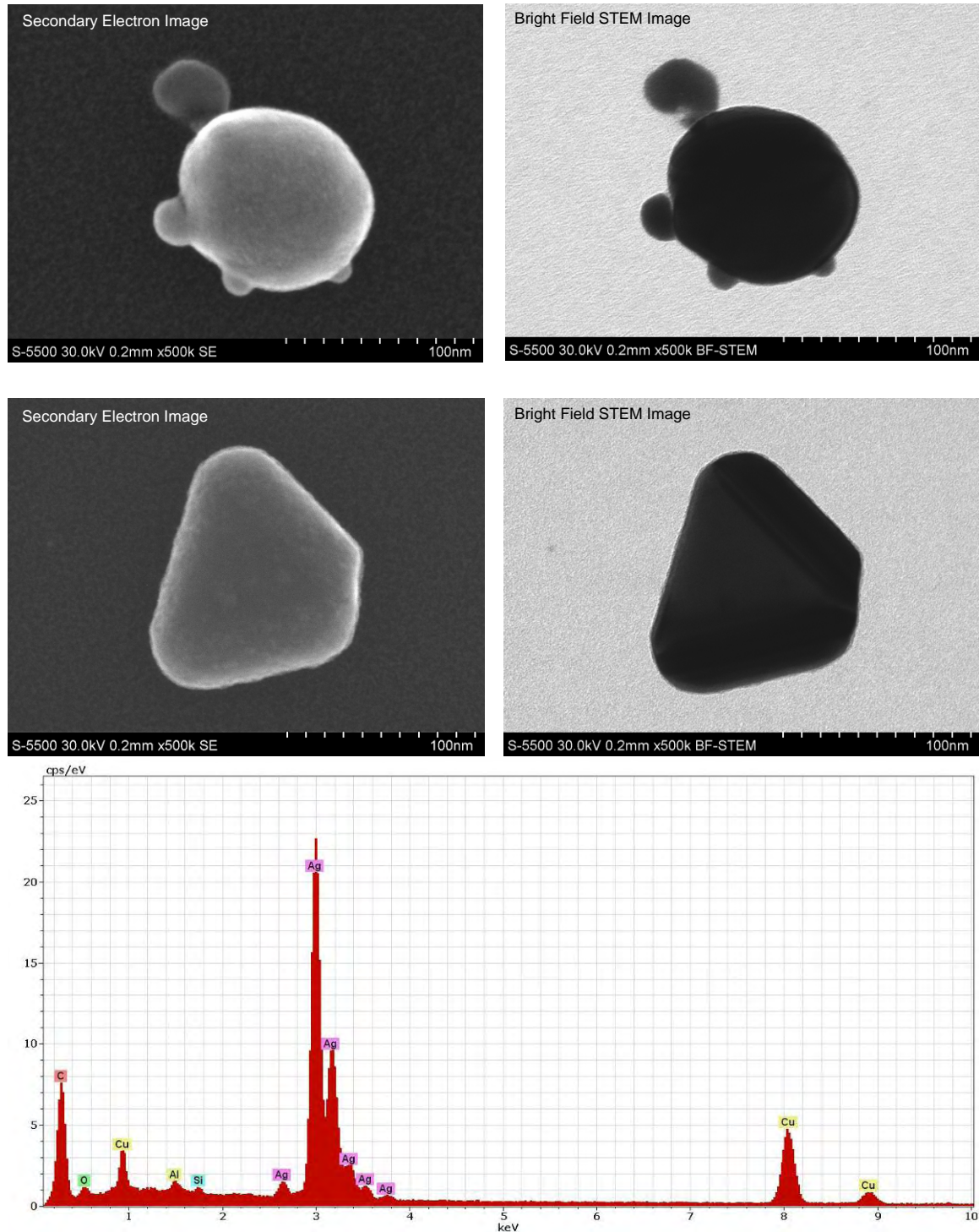


Figure 1. Secondary electron and bright field STEM images along with an elemental spectrum of silver nanoparticles; (Copper (Cu) X-rays are fluoresced from the TEM grid, carbon (C), and aluminum (Al) from the sample holder).

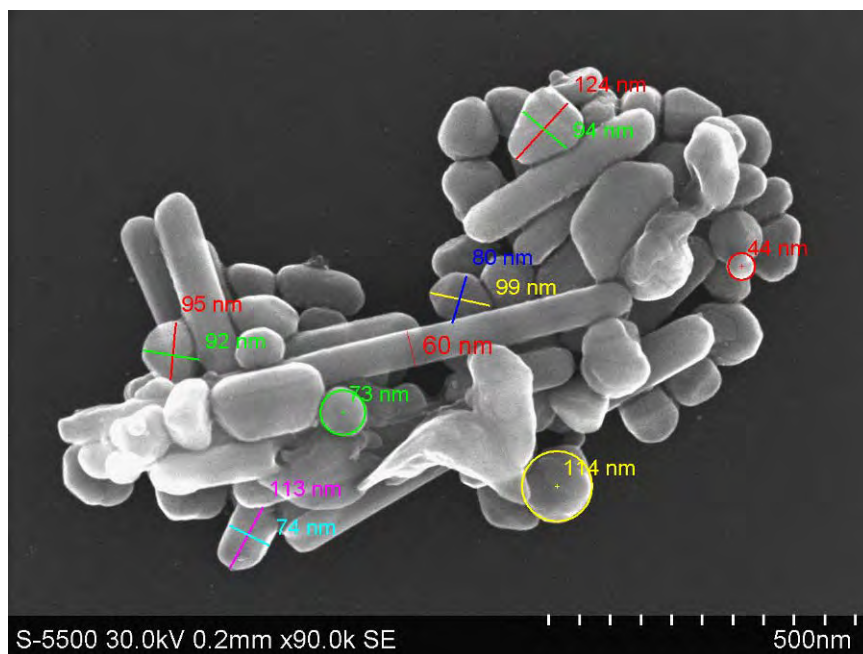


Figure 2a. Secondary electron image illustrating size dimensions of individual particles within an agglomerated structure measuring greater than 1 μm in the longest dimension.

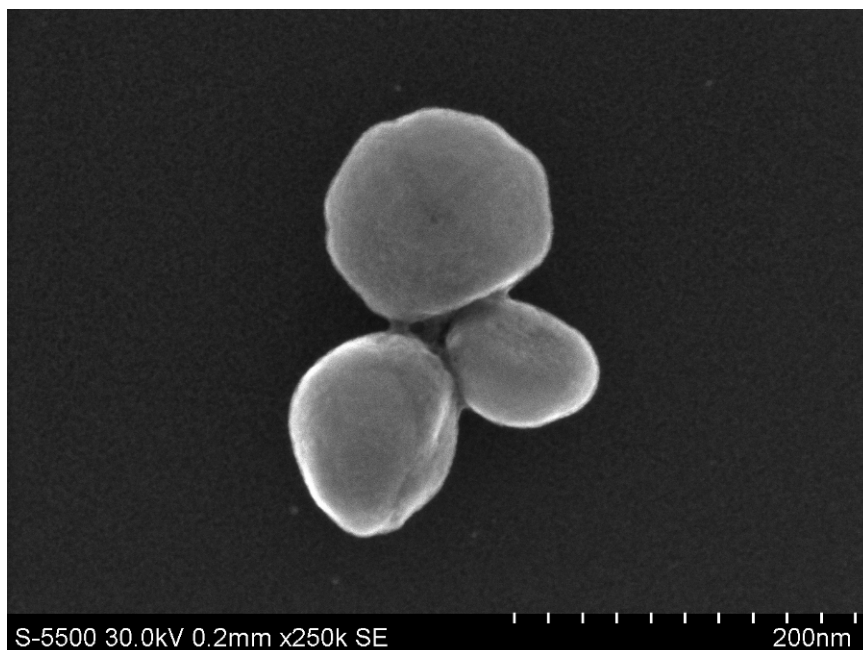


Figure 2b. Secondary electron image of an agglomerated structure comprised of three individual silver particles.

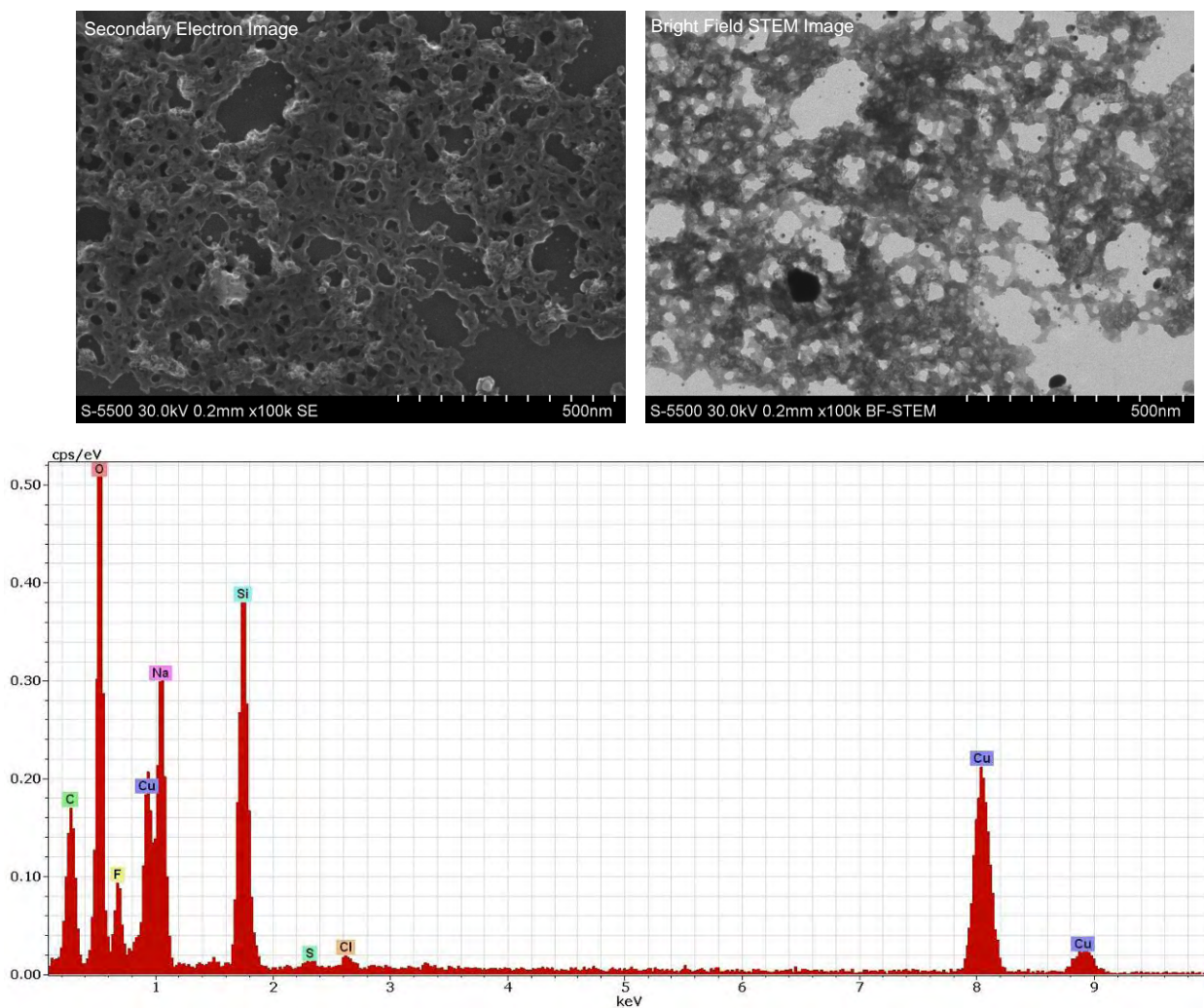


Figure 3. Secondary electron and bright field STEM images along with an elemental spectrum of a residual material associated with the sample. This material consisted primarily of sodium, silicon, and oxygen.

Gold Sample

An aqueous solution (~ 2 ml) containing gold nanoparticles was provided for EM analysis. Sample preparation involved dispersing the particles in the solution using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The EM analysis conducted using the Hitachi HD-2300 revealed that the majority of the sample was comprised of rounded particles that were often spherical. The average particle diameter was 3 nm, although particles were observed down to 1.4 nm. This observation is not in agreement with the MSDS information that indicates a nominal particle size of 30 nm. The EDS analysis of the 3 nm particles confirmed that the material was composed of gold (Au). An EM micrograph and elemental spectrum of the gold particles are presented in Figure 4. Due to the very small size, the gold nanoparticles were only visible in the transmission electron mode. Although not agglomerated, particles had a tendency to cluster on the substrate. Figure 5 and 6 illustrate the particle distribution with size overlays for a number of individual spherical particles.

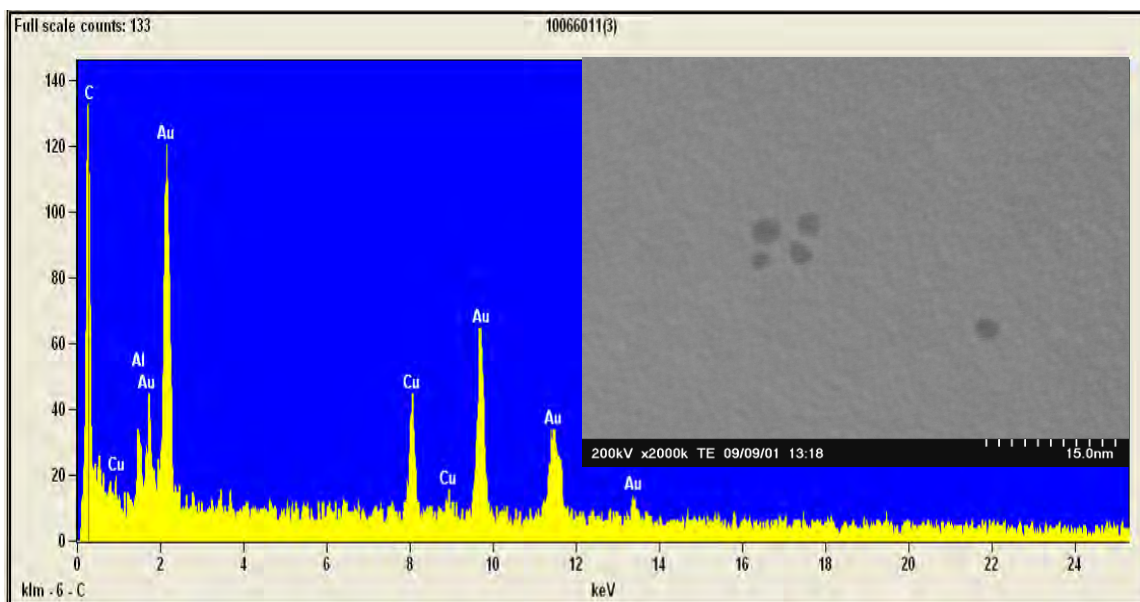


Figure 4. Bright field STEM image along with an elemental spectrum of gold nanoparticles at a magnification of 2,000,000x; (Cu X-rays are fluoresced from the TEM grid, C from the substrate material, and Al from the sample holder).

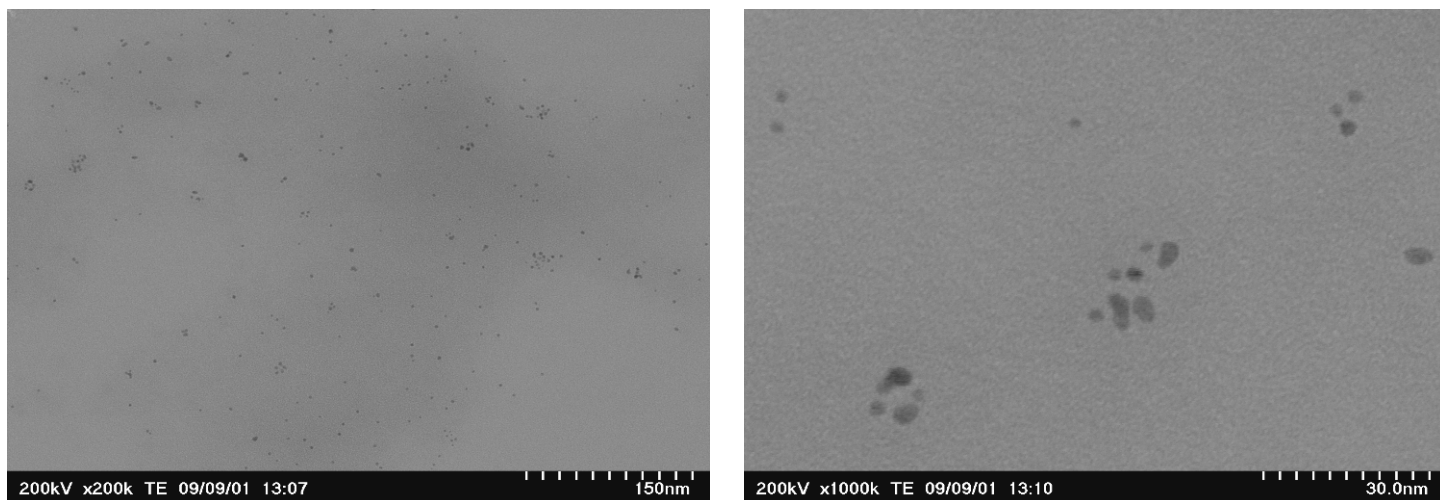


Figure 5. Bright field STEM image showing distribution of Au nanoparticles at 200,000x and 1,000,000x.

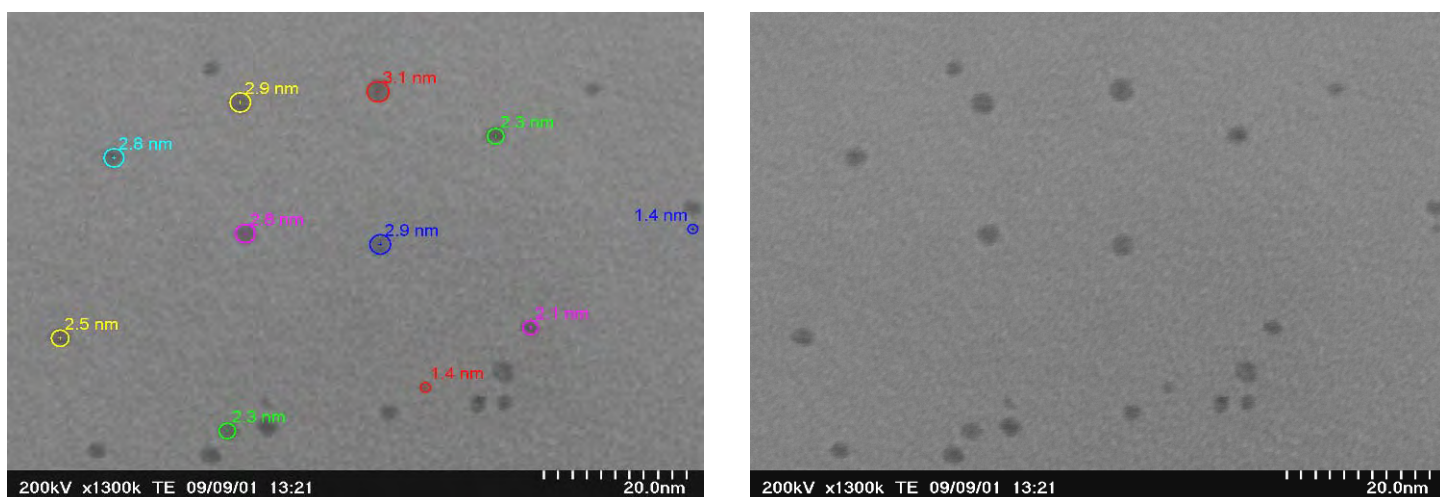


Figure 6. Bright field STEM image showing distribution of Au nanoparticles at 1,300,000x.

Graphene

The graphene dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A lacy Formvar/carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The EM analysis revealed that the majority of the sample was comprised of agglomerated slender platelets. Because individual sheets could not be

distinguished among the agglomerates, size information was difficult to determine. The EM micrograph along with an elemental spectrum presented in Figure 7 illustrates what appears to be an individual platelet on the order of 200 nm in average diameter. The EDS analysis of the material indicated that the carbonaceous material also contained a small amount of silicon and oxygen. Figure 8 provides additional example STEM images illustrating agglomerated structures and the microstructure of the material. The secondary electron images shown in Figure 9 depict a different prospective of the material.

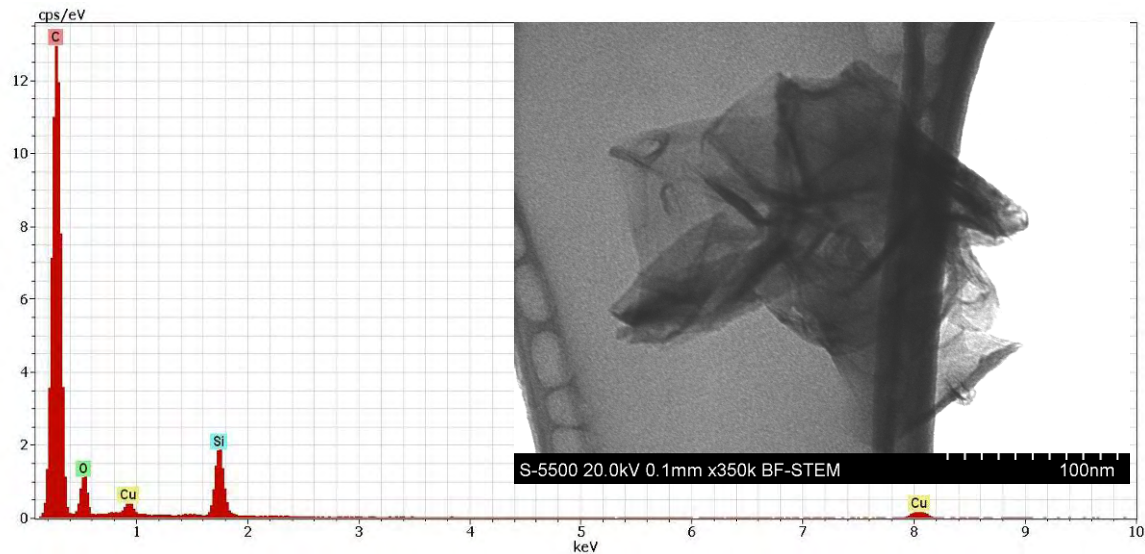


Figure 7. Bright field STEM image along with an elemental spectrum of what appears to be an individual platelet of graphene material.

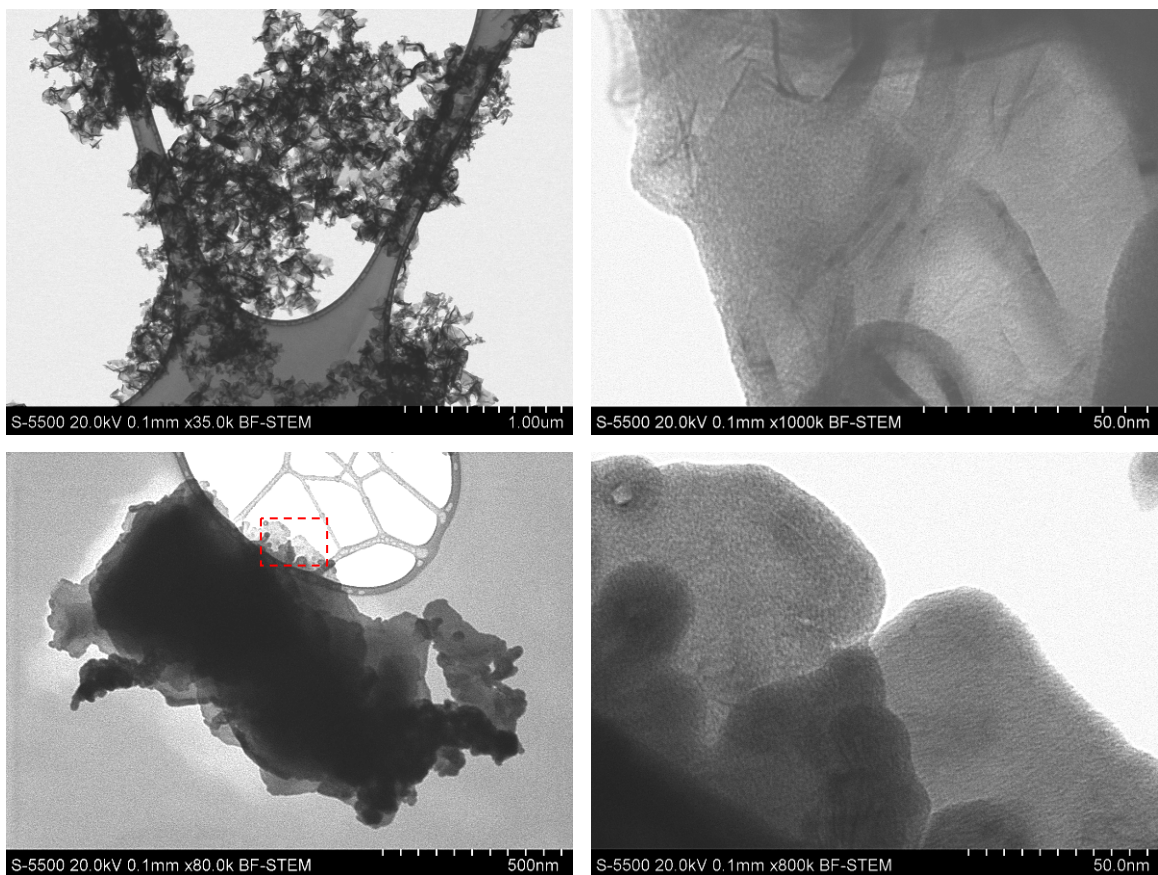


Figure 8. Bright field STEM images illustrating agglomerated graphene material and platelet microstructure.

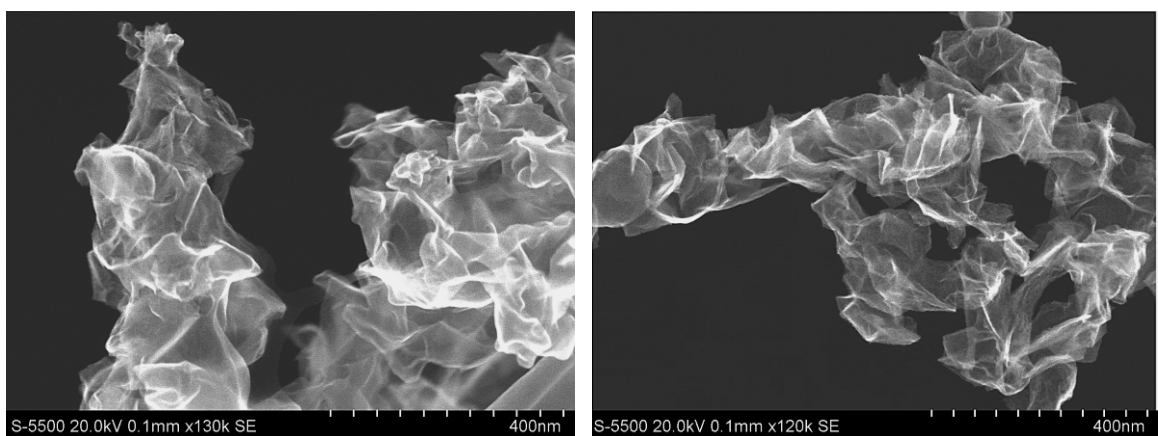


Figure 9. Secondary electron images illustrating agglomerated graphene material.

Acetylene Black

The acetylene black dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A lacy Formvar/carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The sample was comprised of aciniform structures consisting of agglomerated irregularly shaped primary particles that were not well defined per ASTM 6602-03b². Although individual nodules were difficult to distinguish, primary particle size appeared to be consistent averaging approximately 30-40 nm in diameter. The size of the agglomerates was variable measuring on the order of micrometers. EDS analysis of the material confirmed that the particles were composed of carbon. EM micrographs and an elemental spectrum of aciniform structures are presented in Figure 10. Figure 11 illustrates the primary particles at an increased magnification. The microstructure of the material as shown in Figure 12 revealed an outer shell in the primary particles; however the inner layers are less ordered compared to the carbon black sample.

² ASTM International Designation: D 6602 – 03b; Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both¹; ASTM Committee D24; Published January 2004.

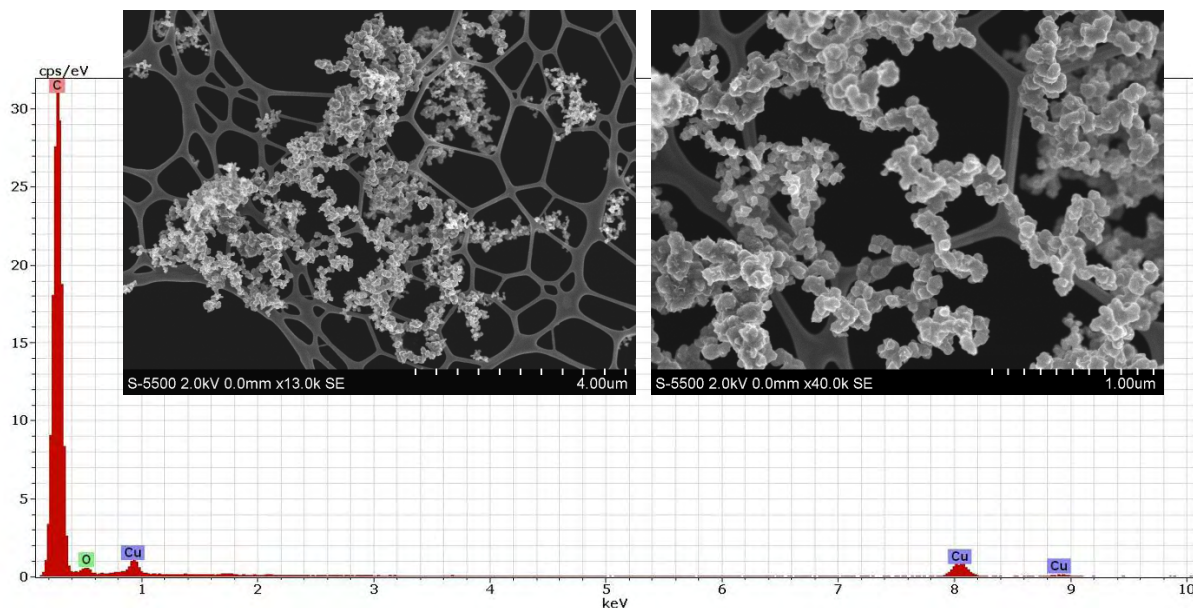


Figure 10. Secondary electron images and elemental spectrum of acetylene black.

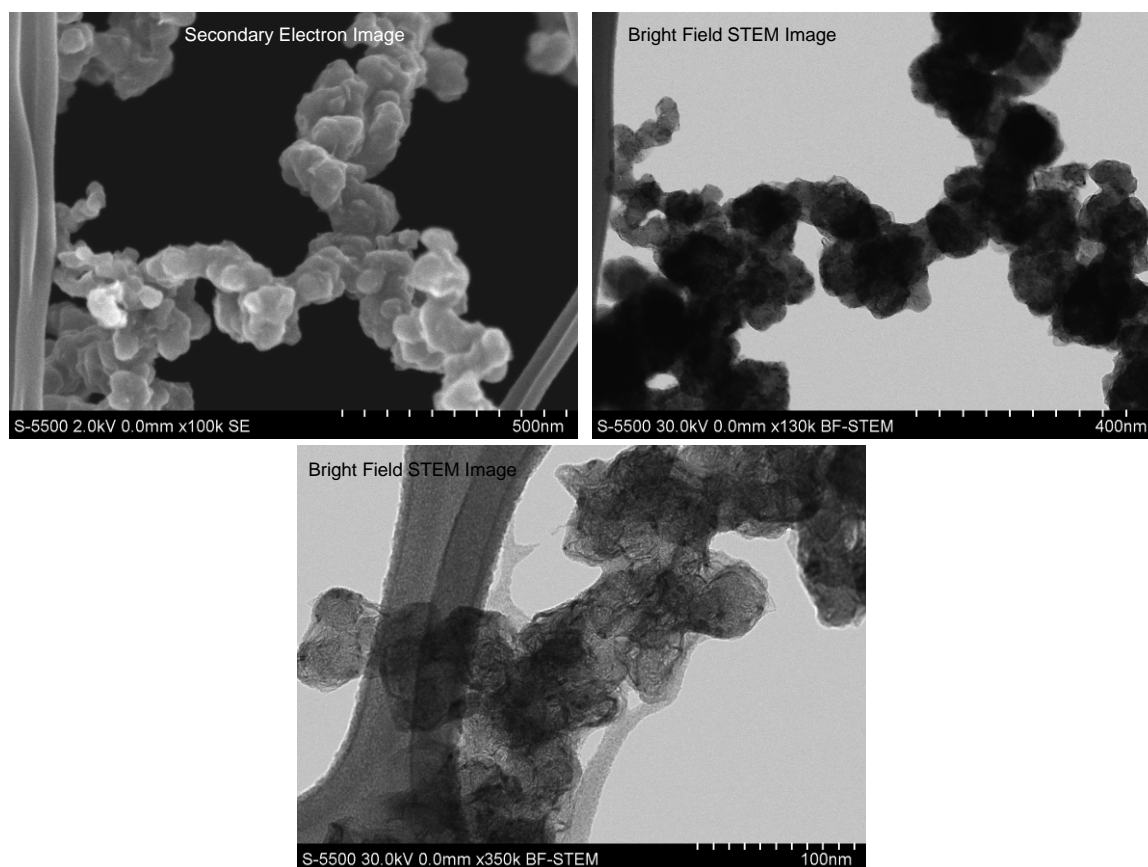


Figure 11. Secondary electron and bright field STEM images of acetylene black.

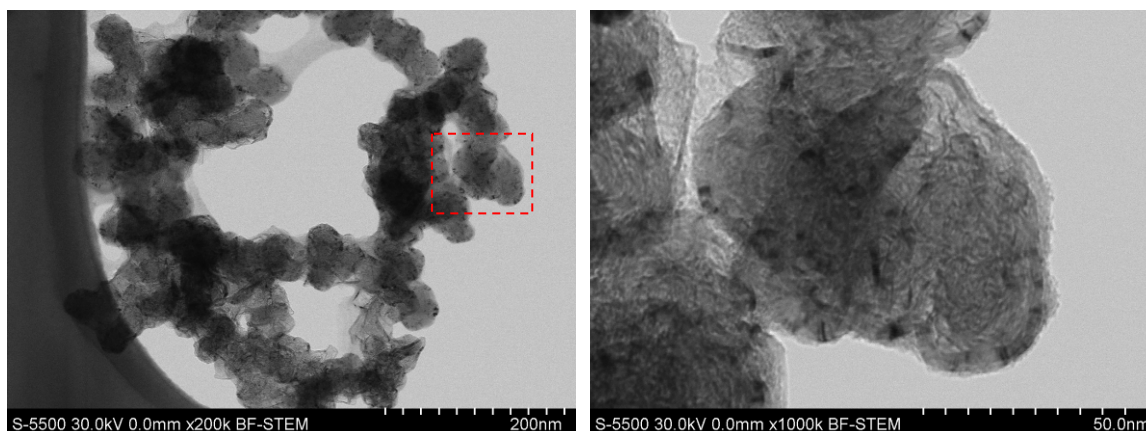


Figure 12. Bright field STEM images illustrating primary particle microstructure of acetylene black.

Carbon Black

The carbon black dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A lacy Formvar/carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The sample was comprised of aciniform structures consisting of agglomerated irregular and rounded primary particles that averaged approximately 50-75 nm in diameter. The size of the agglomerates was variable measuring on the order of micrometers. The EDS analysis of the material confirmed that the particles were composed of carbon. Primary particles were well defined with a smooth particle perimeter per ASTM 6602- 03b. EM micrographs and an elemental spectrum of the aciniform structures are presented in Figure 13. Figure 14 illustrates the primary particles at an increased magnification. The microstructure of the material as shown in Figure 15 revealed an outer shell with multiple sets of inner concentric layers.

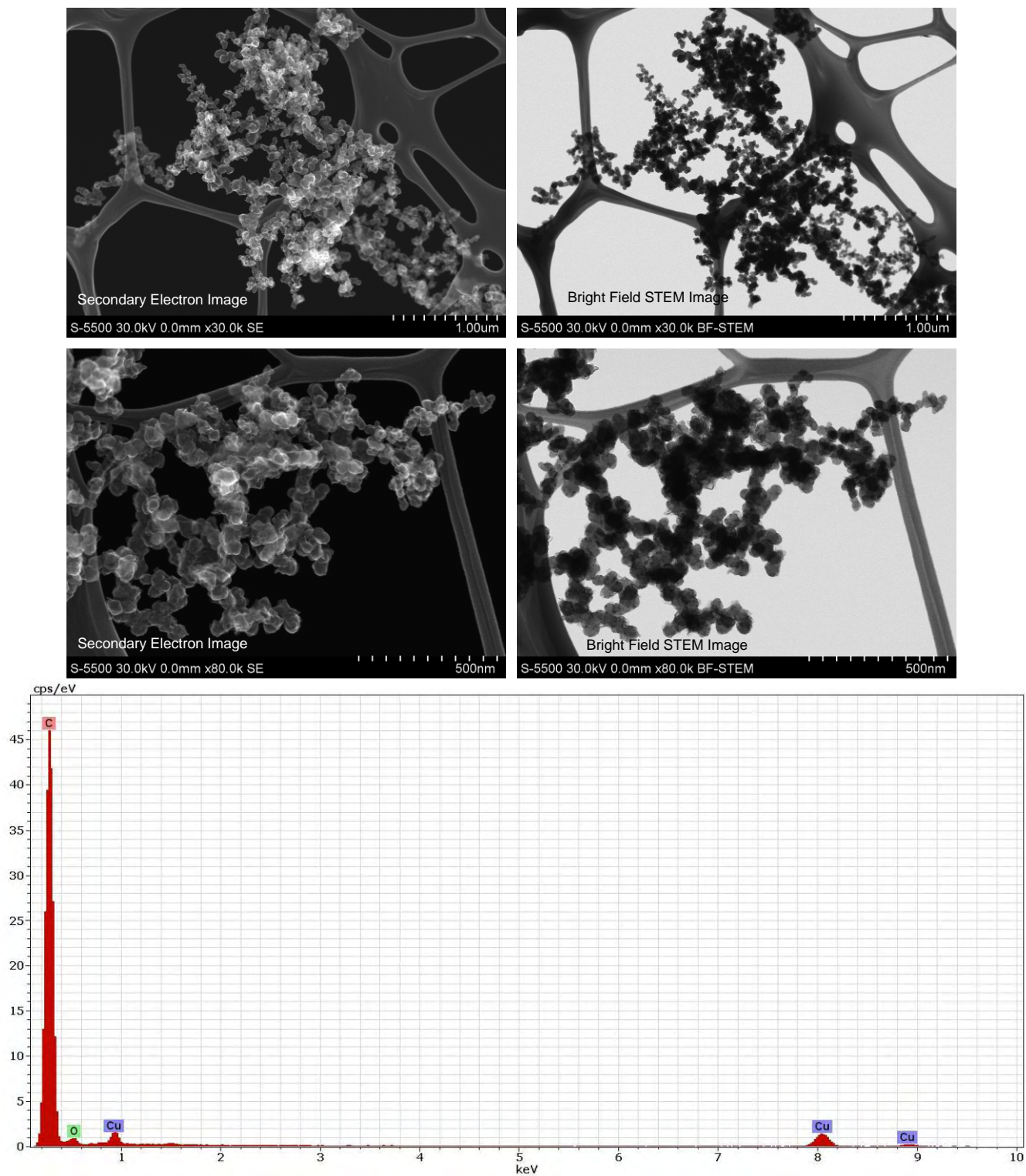


Figure 13. Secondary electron and bright field STEM images and elemental spectrum of carbon black.

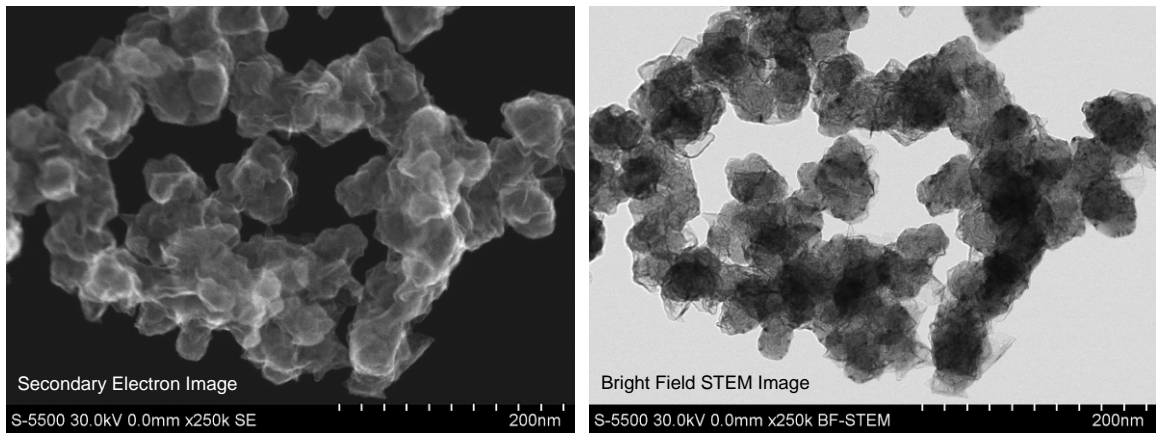


Figure 14. Secondary electron and bright field STEM images of carbon black.

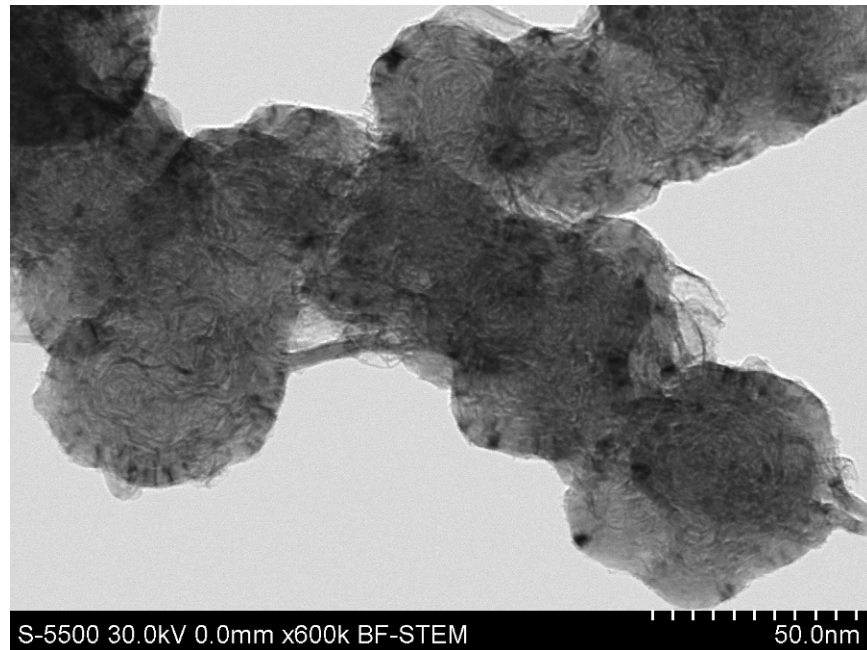


Figure 15. Bright field STEM image illustrating primary particle microstructure of carbon black.

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Should you have any questions regarding this information, please do not hesitate to contact us.

Sincerely,

A handwritten signature in cursive script, reading "Kristin L. Bunker".

Kristin L. Bunker, Ph.D.
Senior Scientist

cc: G. Casuccio (RJLG)



September 18, 2009

Ms. Linnea Wahl
Pilot Study Project Manager
Ernest Orlando Lawrence Berkeley National Laboratory
Berkeley, CA 94720

Re: Electron Microscopy Results of Potential UNP
PI: Vincent Battaglia: Building 70, Lab 295/299

Dear Linnea:

This report summarizes the electron microscopy (EM) results for the analysis of the samples collected from Building 70, Lab 295/299 as part of the Phase I pilot study related to potential unbound engineered nanoparticle (UNP) releases. Research conducted in this laboratory is directed by Vincent Battaglia and focuses on battery materials. Nanomaterials being utilized in these research areas include carbon (acetylene) black, lithium titanate, lithium iron phosphate, and silicon. Information related to the samples is listed in Table 1. The EM analysis was performed to provide information on particle size, morphological characteristics, and elemental composition associated with each material.

Table 1: Sample Information

Client Sample ID	RJLG Sample #	Sample Received Date	Sample Analysis Date	Analyst
Carbon (Acetylene) Black	10066035	August 5, 2009	September 2, 2009	Kristin Bunker
LithiumTitanate Nanopowder	10066036	August 5, 2009	September 1, 2009	Kristin Bunker
Lithium Iron Phosphate Nanoparticles	10066037	August 5, 2009	September 2, 2009	Kristin Bunker
Silicon Nanopowder	10066038	August 5, 2009	September 1, 2009	Kristin Bunker

The samples were characterized using an Hitachi S-5500 high resolution field emission scanning electron microscope (HR-FESEM) with scanning transmission electron microscopy (STEM) capabilities. The analysis was conducted primarily in the secondary and bright field transmission electron modes at magnifications up to 1,000,000x. Compositional information was obtained through collection and processing of characteristic X-rays using a Bruker energy dispersive X-ray spectroscopy (EDS) system incorporating a 30 mm² silicon drift detector (SDD).

The lithium titanate, lithium iron phosphate, and silicon samples were analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES). A Varian 730ES ICP-OES was used to detect and quantify the elements in the material including trace metals by measuring light emitted at wavelengths characteristic of individual elements. The intensity of this emission is indicative of the concentration of the element within the sample.

The samples were prepared for ICP-OES using acid digestion techniques. The limited sample size and the unique composition of the samples did not allow for the preparation/digestion of any matrix duplicate (MD), matrix spike (MS) and laboratory control standard (LCS) quality control samples. A laboratory reagent blank (LRB) along with a fortified laboratory reagent blank (LFB) were prepared and analyzed instead. Elevated silicon (1.864 mg/L) in the LRB results was due to etching of the glass in the ICP-OES system by the hydrofluoric acid (HF) used to digest the samples. Elevated phosphorus (0.055 mg/L) in the LRB results was due to the sample container used to perform the digestion. All of the recoveries reported in the LFB results were within the acceptable laboratory limits ($\pm 15\%$), with the exception of silicon (for the same reasons as described above for the LRB) and calcium (which tends to form insoluble CaF_2 in the presence of HF). A detailed description of the sample preparation is described along with the results of the analysis in the table attached to this report.

The table below (Table 2) provides a concise summary of the EM analysis results. The sections following the table provide a summary of the EM results for the carbon (acetylene) black, lithium titanate, lithium iron phosphate, and silicon samples.

Table 2: Sample Attributes

	Particle Size	Morphology	Chemistry
Carbon (Acetylene) Black	Primary particles ~30-40 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of rounded and irregularly shaped particles	SEM/EDS: C
Lithium Titanate Nanopowder	Individual particles ranged from 50 nm to >5 μ m; Agglomerates on the order of micrometers	Irregular shaped agglomerated particles	SEM/EDS: Ti, O and trace of Si; Trace of Ti-O-Zr ICP-OES: Major elements include Ti, Li, Si and trace amounts of Zr, Na, P, Fe, Al, Ni
Lithium Iron Phosphate Nanoparticles	Particles average >100 nm, ranging from ~50 nm to 500 nm; rod-shaped particles measured ~100-200 nm in diameter; Agglomerates on the order of micrometers	Rounded and rod-shaped particles; trace amount of angular particles	SEM/EDS: Fe, P, and O ICP-OES: Major elements include Fe, P, Li and trace amounts of Mn, Si, S, Al, Cr, K, Zn, Na
Silicon Nanopowder	Primary particles ~10-50 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of rounded particles, often spherical	SEM/EDS: Si, O ICP-OES: Major element was Si and trace amounts of Na, P, B

Carbon (Acetylene) Black

The carbon (acetylene) black dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A lacy Formvar/carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The sample was comprised of aciniform structures consisting of agglomerated irregular and rounded primary particles that averaged approximately 30-40 nanometers (nm) in diameter. The size of the agglomerates was variable measuring on the order of micrometers. Primary particles were well defined overall with a smooth particle perimeter per ASTM 6602- 03b. The EDS analysis of the material confirmed that the particles were composed of carbon. EM micrographs and an elemental spectrum of the aciniform structures are presented in Figure 1. Figure 2 illustrates the primary particles at an increased magnification. The microstructure of the material as shown in Figure 3 revealed an outer shell with inner concentric layers.

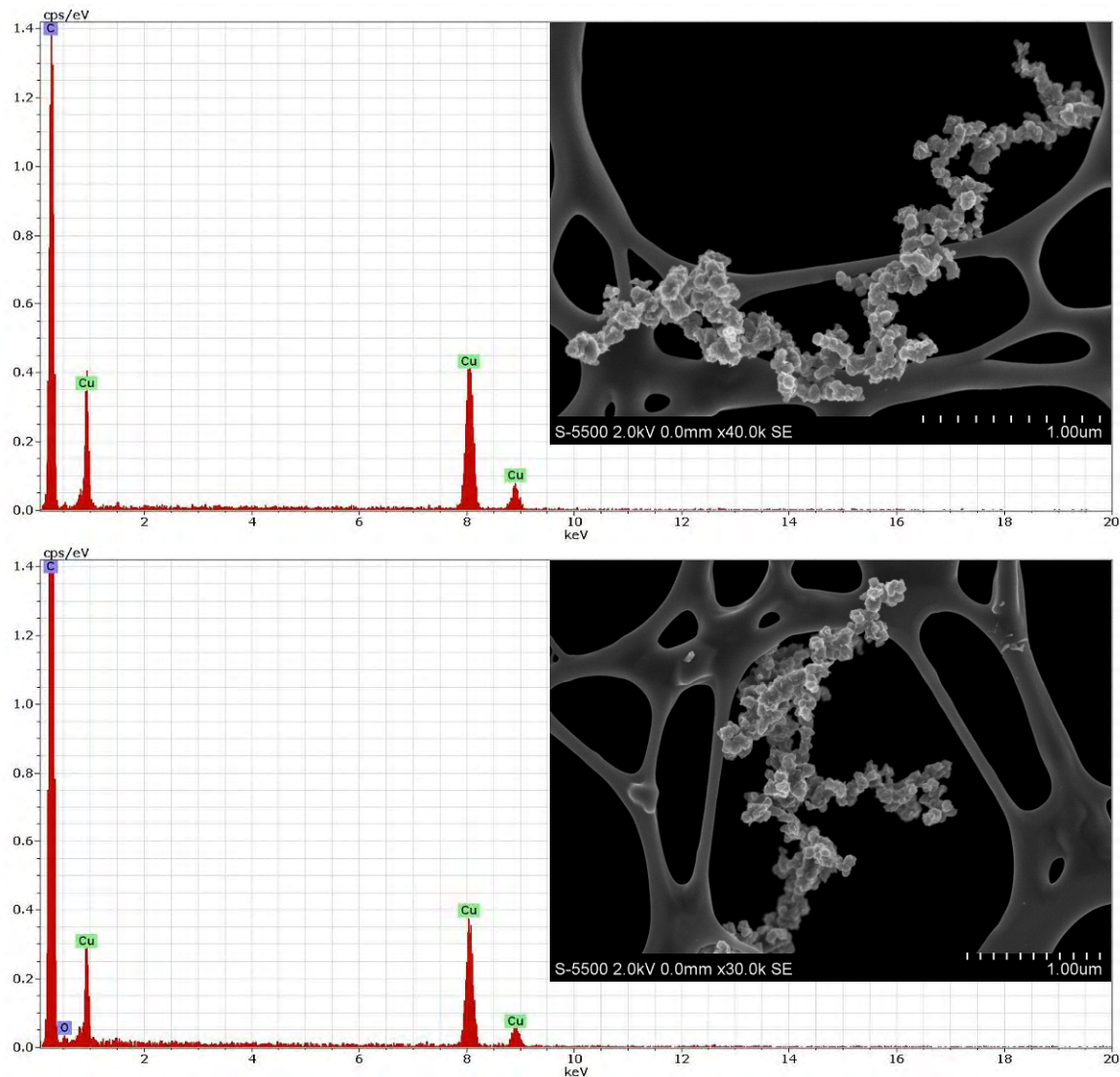


Figure 1. Secondary electron images and elemental spectra of carbon (acetylene) black; (Copper (Cu) X-rays are fluoresced from the TEM grid).

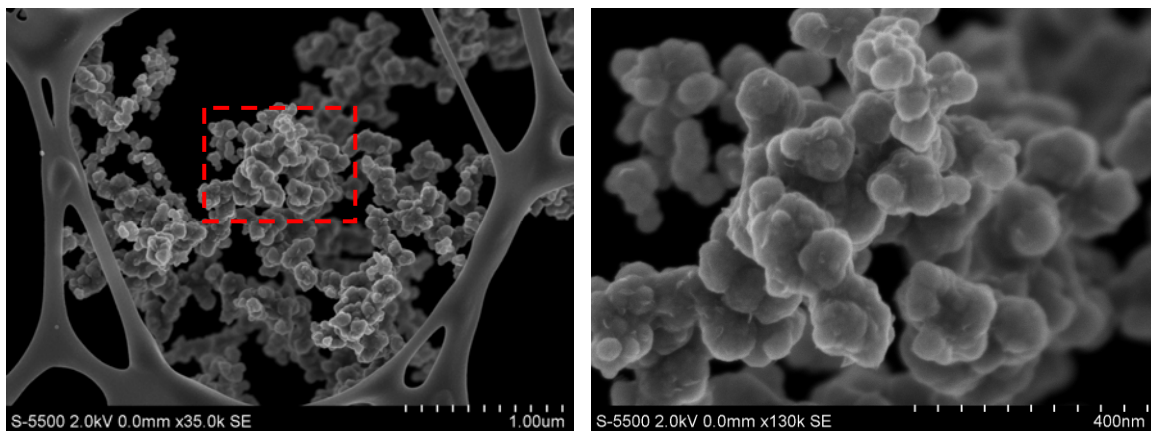


Figure 2. Secondary electron images of carbon (acetylene) black.

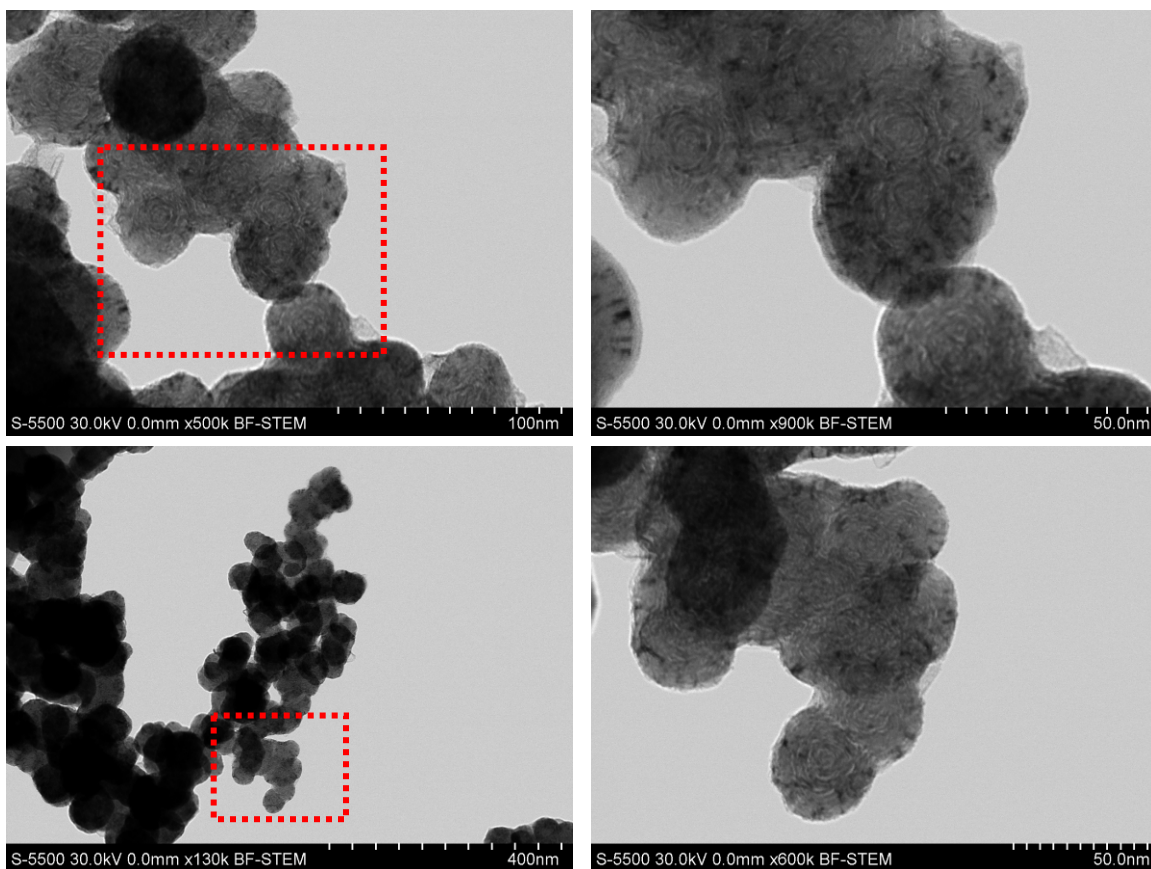


Figure 3. Bright field STEM images of carbon (acetylene) black. High magnification images on the right illustrate the microstructure of the primary particles.

Lithium Titanate Powder

The lithium titanate dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The majority of the sample was comprised of irregular shaped agglomerates with rough surfaces in the micrometer size range. Individual particles ranged from 50 nm to greater than 5 micrometers (μm). Figure 4 illustrates the morphological characteristics of the material. EDS analysis indicated that the sample was comprised of titanium (Ti) and oxygen (O) with trace amounts of silicon (Si), while lithium can not be detected using EDS analysis. EM images and elemental spectra are provided in Figure 5. The images shown in Figure 5 illustrate the diversity in size. One Ti-O structure contained a trace of zirconium (Zr). The EDS and X-ray map shown in Figure 6 was collected from the particle containing a trace of Zr. The X-ray data indicates that the Zr was localized in one region of the particle. Figure 7 illustrates a residual material that was associated with the sample. EDS analysis of the residue revealed that the material was comprised of carbon (C) with a trace oxygen (O) and silicon (Si).

The lithium titanate sample was also analyzed by ICP-OES. The sample was comprised of Ti, lithium (Li), Si and trace amounts of Zr, sodium (Na), phosphorus (P), iron (Fe), aluminum (Al), and nickel (Ni). The results can be found at the end of the report.

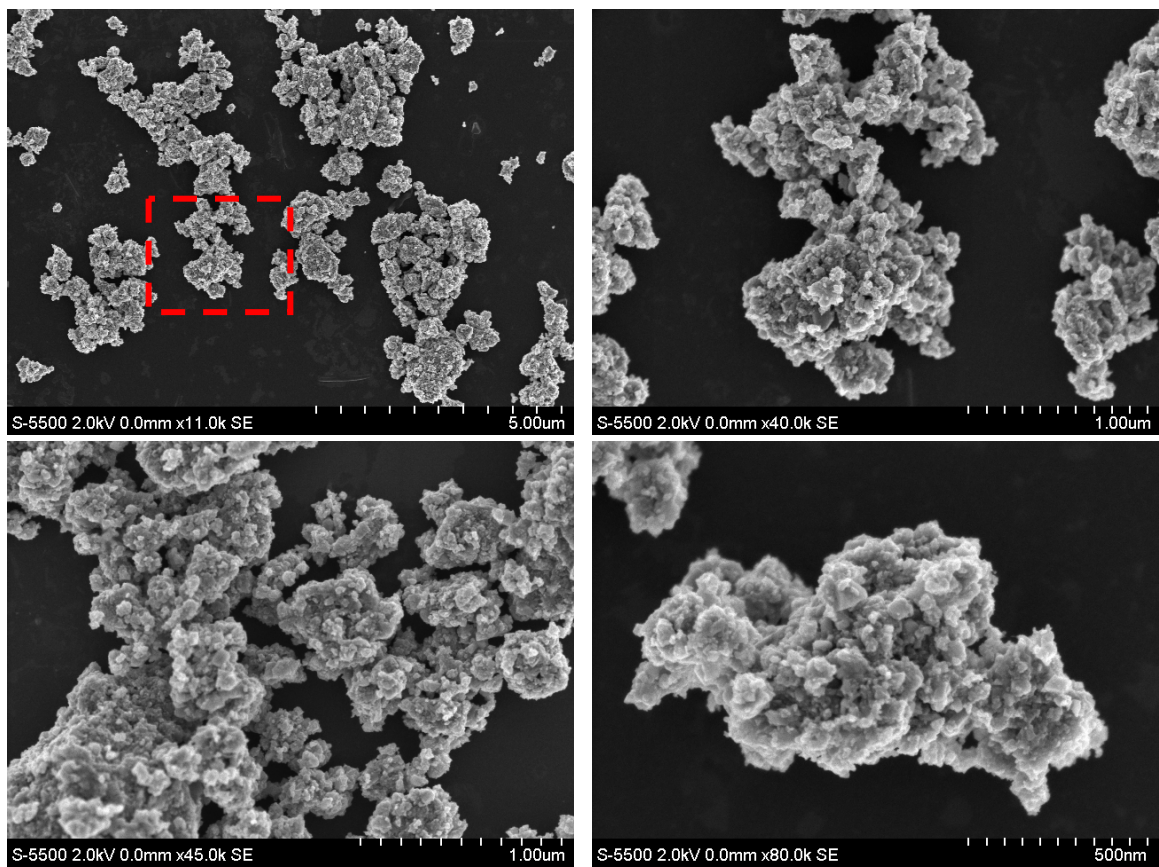


Figure 4. Secondary electron images of lithium titanate acquired at an accelerating voltage of 2kV.

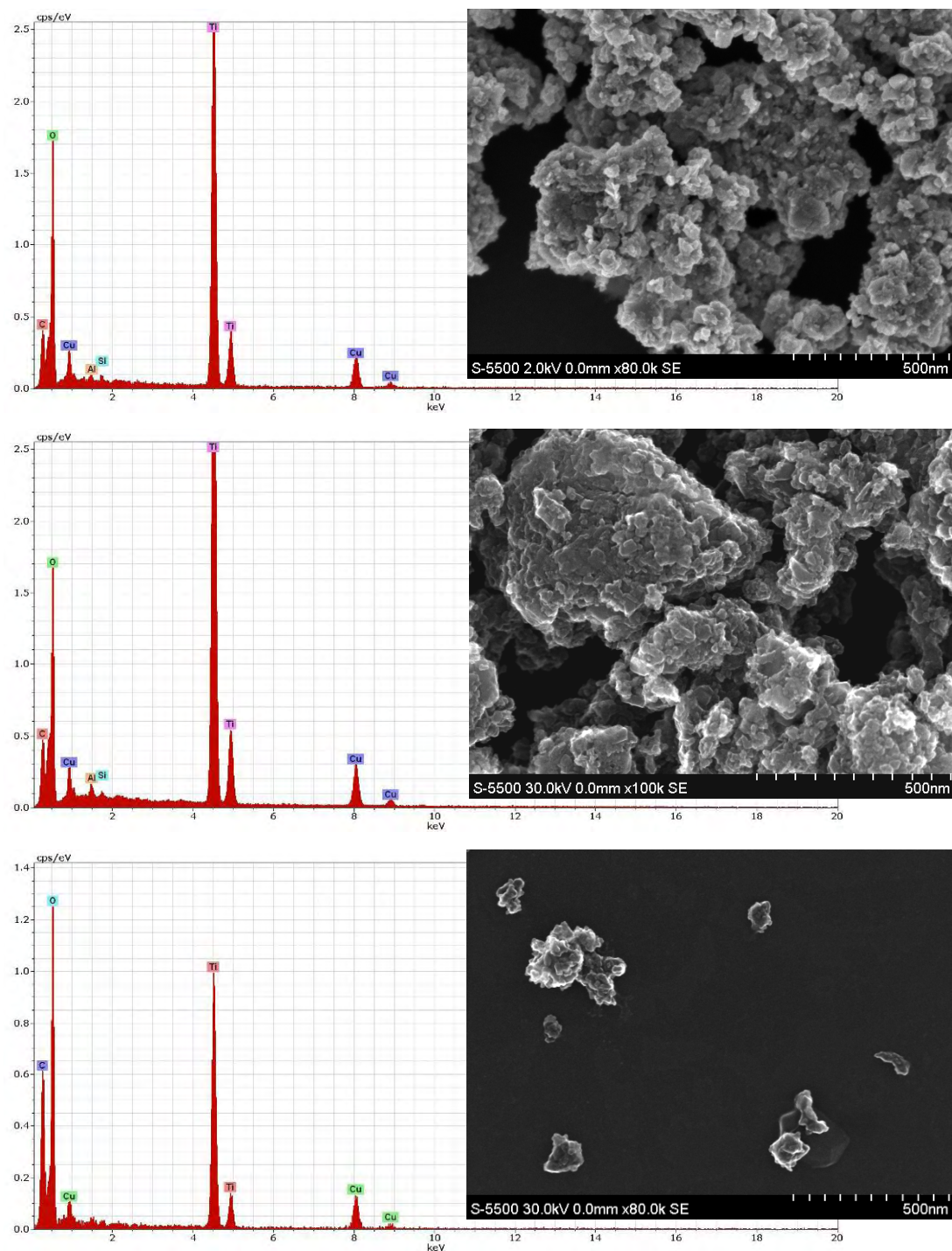


Figure 5. Secondary electron images and elemental spectra of lithium titanate. The top (2 kV) and middle (30 kV) images illustrate the material using different accelerating voltages. The lower accelerating voltage (2 kV) provides finer surface detail on the particles. (Copper (Cu) X-rays are fluoresced from the TEM grid, carbon (C) from the substrate material, and Al from the sample holder).

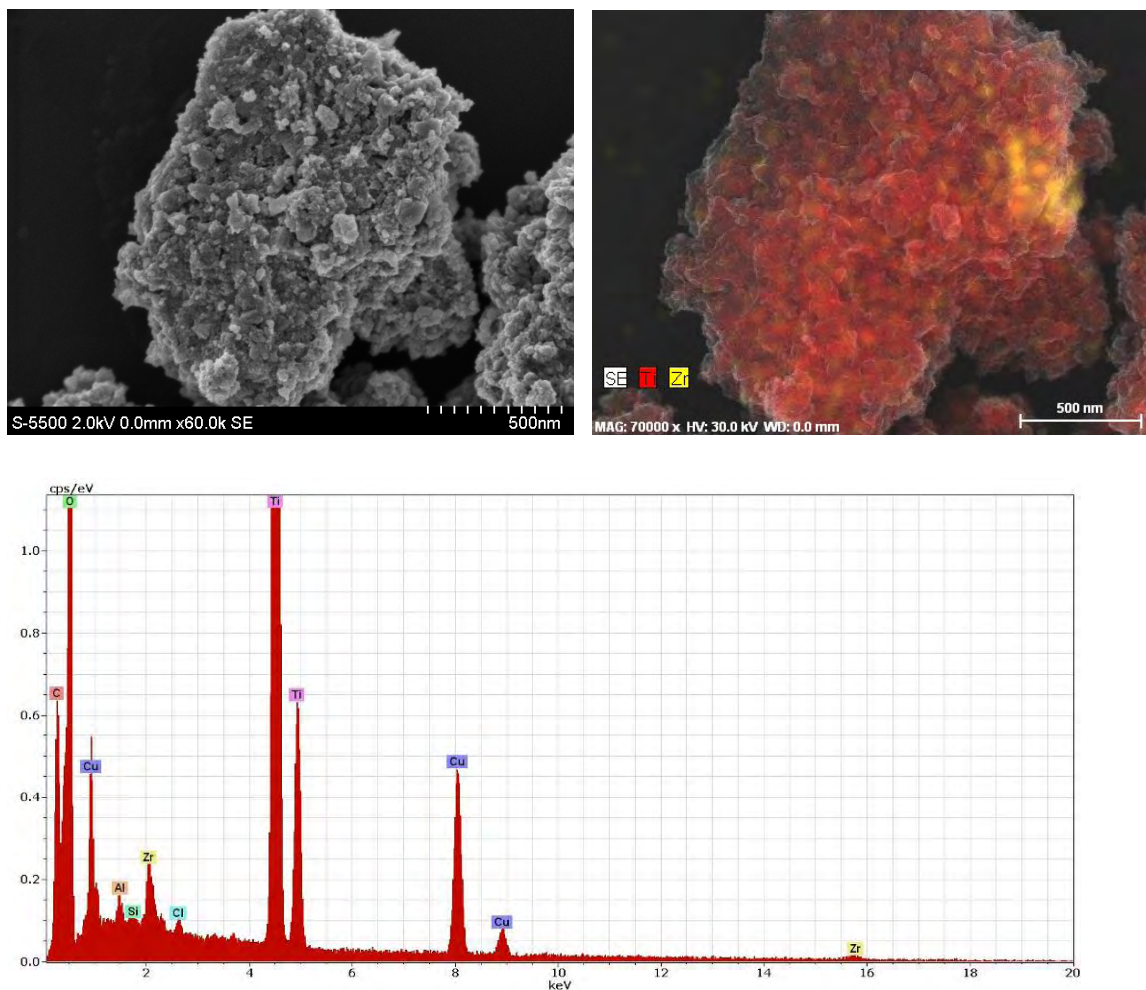


Figure 6. Secondary electron image (left) of a structure containing zirconium localized in one region of the particle as shown by the composite X-ray map (right). X-ray maps visually depict the number of x-rays for the elements being mapped at each pixel. Particle phases discriminated by variations in elemental composition are shown in the color variations (i.e., Zr in yellow and Ti in red).

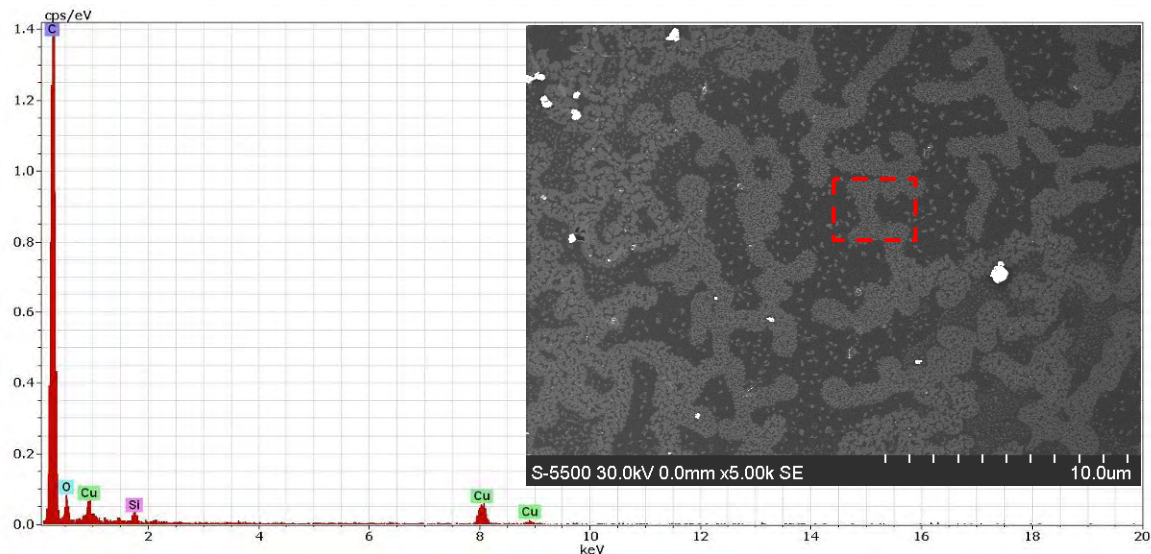


Figure 7. Secondary electron images and elemental spectrum of the residual material associated with the sample.

Lithium Iron Phosphate

The lithium iron phosphate dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The EM analysis revealed that the sample was comprised primarily of individual rounded particles and rod-shaped particles with rounded ends. The majority of the particles measured greater than 100 nm, ranging from approximately 50-500 nm in average diameter. Rod-shaped particles measured ~100-200 nm in diameter. For a small number of particles, an angular shape was observed. Rounded, angular, and rod-shaped particles each had a smooth surface texture and were often agglomerated. Agglomerated structures measured on the order of micrometers. EDS analysis indicated that the material was composed of iron (Fe), phosphorous (P), and oxygen (O), while lithium can not be detected using EDS analysis. EM images and elemental spectra are provided in Figure 8. Additional EM images are provided in Figures 9 and 10 illustrating variability in agglomerate size. A platelet material was associated with the material, discernible in the higher magnification images.

The lithium iron phosphate sample was also analyzed by ICP-OES. The sample was comprised Fe, P, lithium (Li) and trace amounts of manganese (Mn), silicon (Si), sulfur (S), aluminum (Al), chromium (Cr), potassium (K), zinc (Zn), sodium (Na). The results can be found at the end of the report.

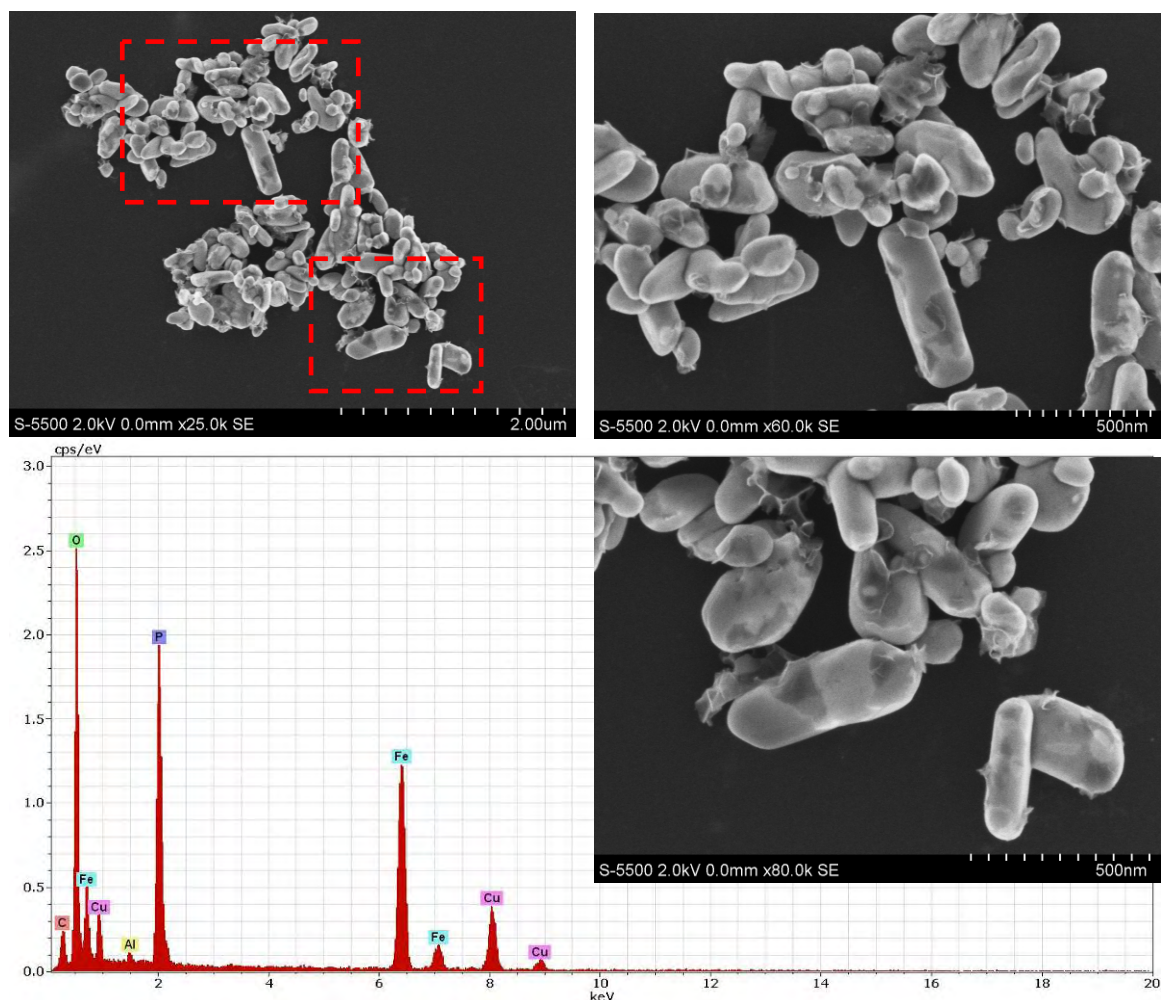


Figure 8. Secondary electron images and elemental spectrum of lithium iron phosphate.

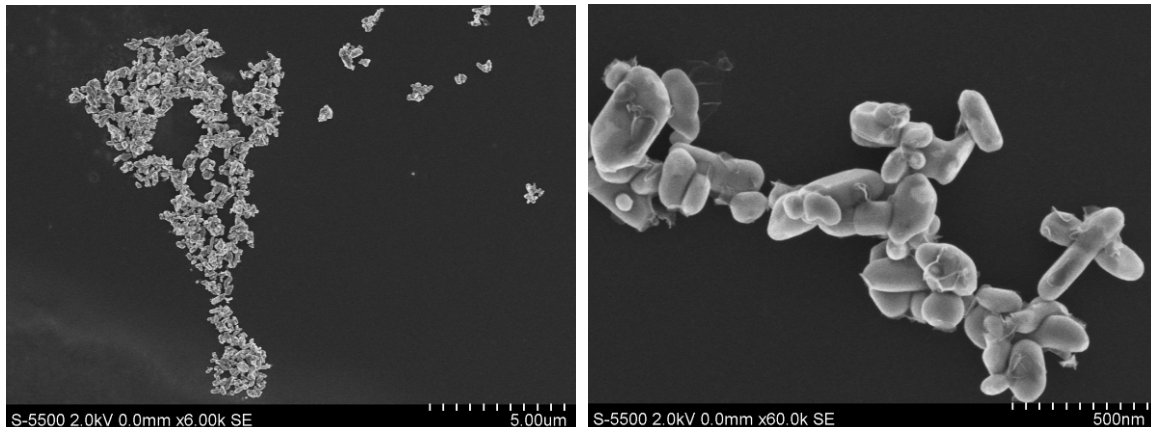


Figure 9. Secondary electron images of lithium iron phosphate.

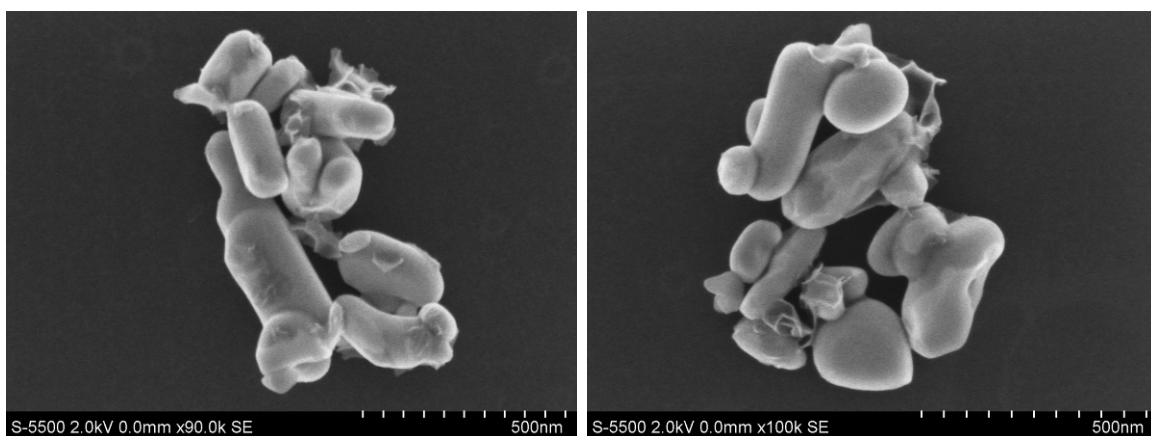


Figure 10. Secondary electron images of lithium iron phosphate.

Silicon Nanopowder

The silicon dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample preparation was dried on a hot plate for 5 minutes.

The sample was comprised of rounded, often spherical, agglomerated particles with a smooth surface texture and aciniform characteristics. Primary particle size ranged from approximately 10 to 50 nm in diameter, although agglomerations measured on the order of micrometers. The EDS analysis of the material confirmed that the particles were composed of silicon and oxygen. EM micrographs and an elemental spectrum are presented in Figures 11 and 12. Figure 13 illustrates the primary particles at an increased magnification. There

was a C-rich residue with trace amounts of Si, Na, and Ca on the substrate. The residue is visible in the background of the image shown in Figure 11.

The silicon sample was also analyzed by ICP-OES. The sample was comprised of Si and trace amounts of Na, phosphorus (P), and boron (B). The results can be found at the end of the report.

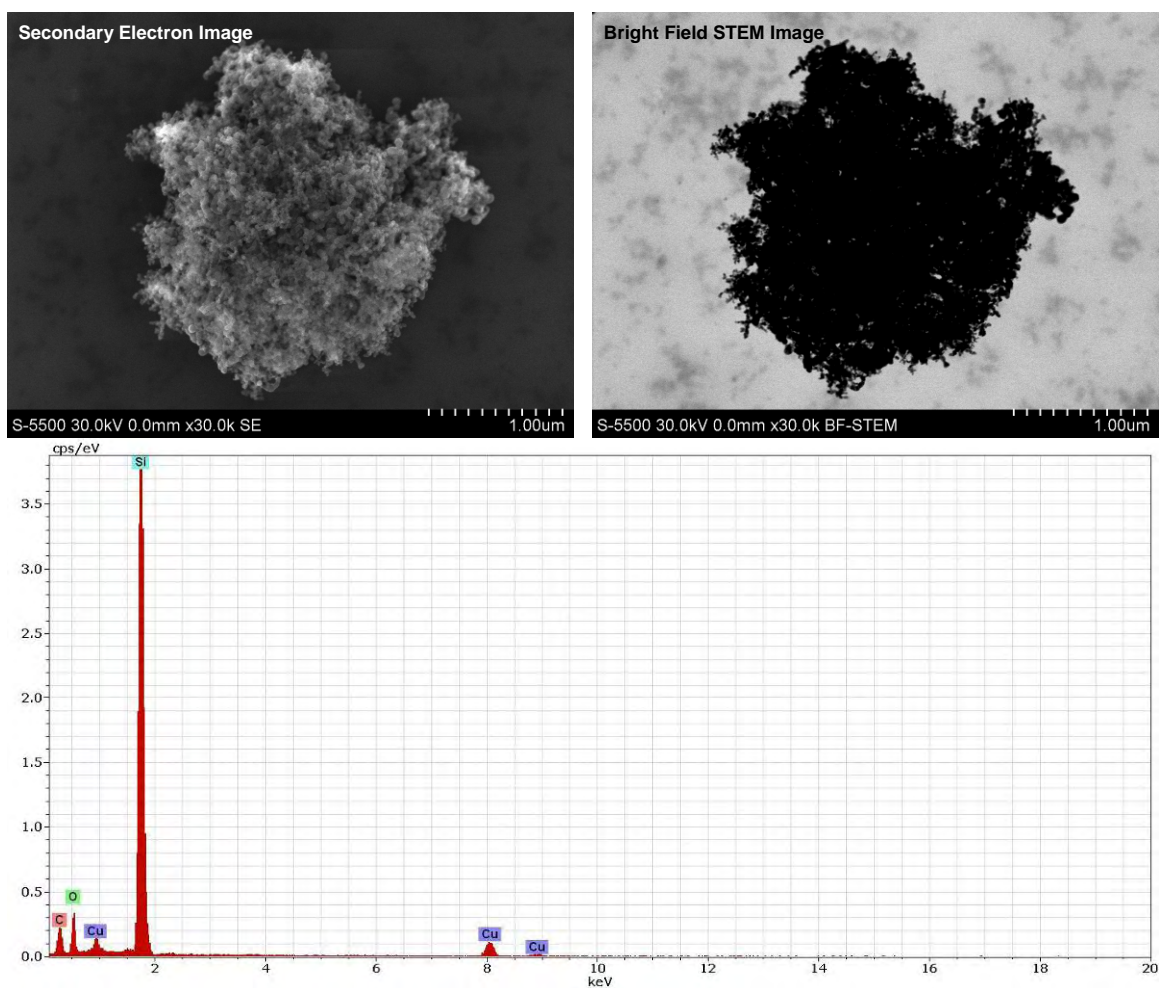


Figure 11. Secondary electron and bright field STEM images and elemental spectrum of the silicon nanopowder.

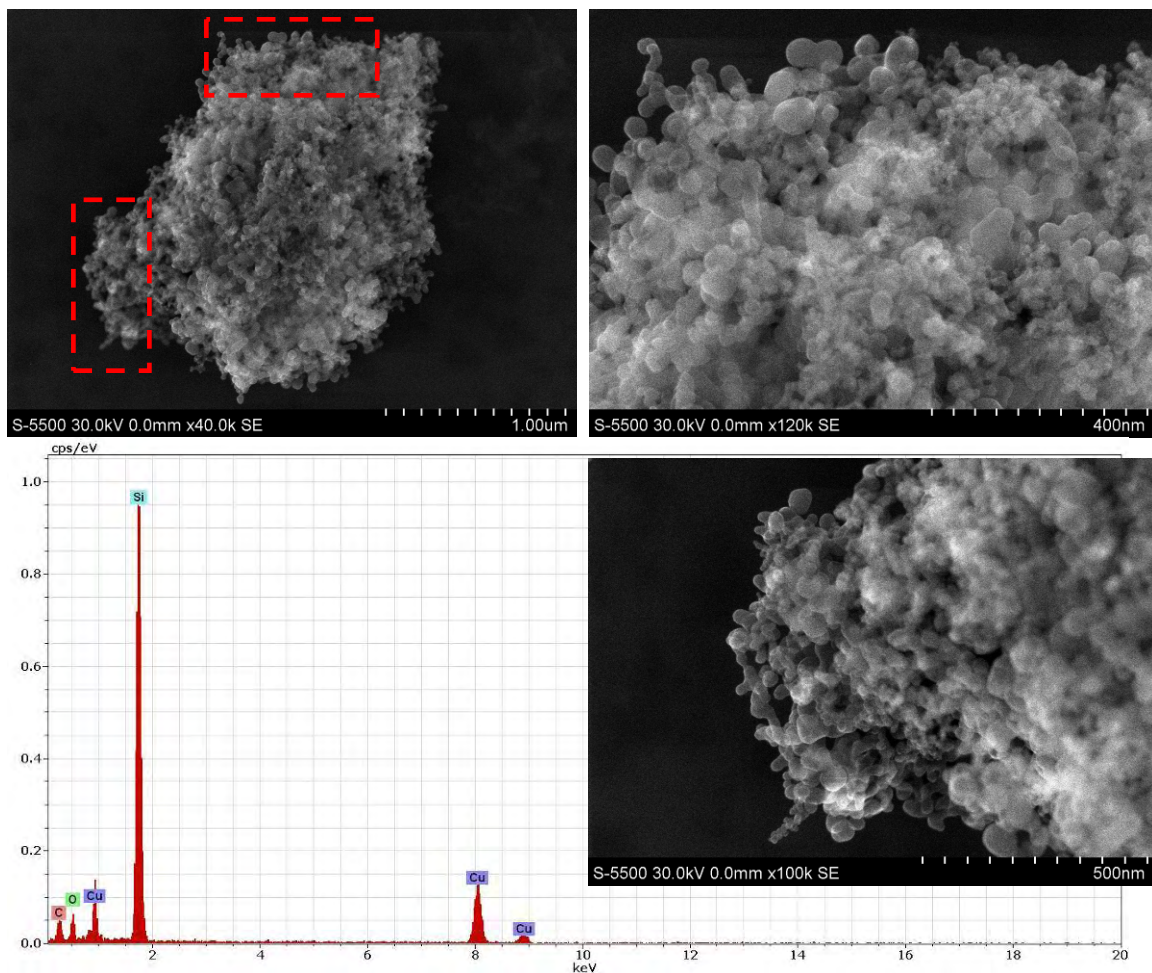


Figure 12. Secondary electron and bright field STEM images and elemental spectrum of the silicon nanopowder.

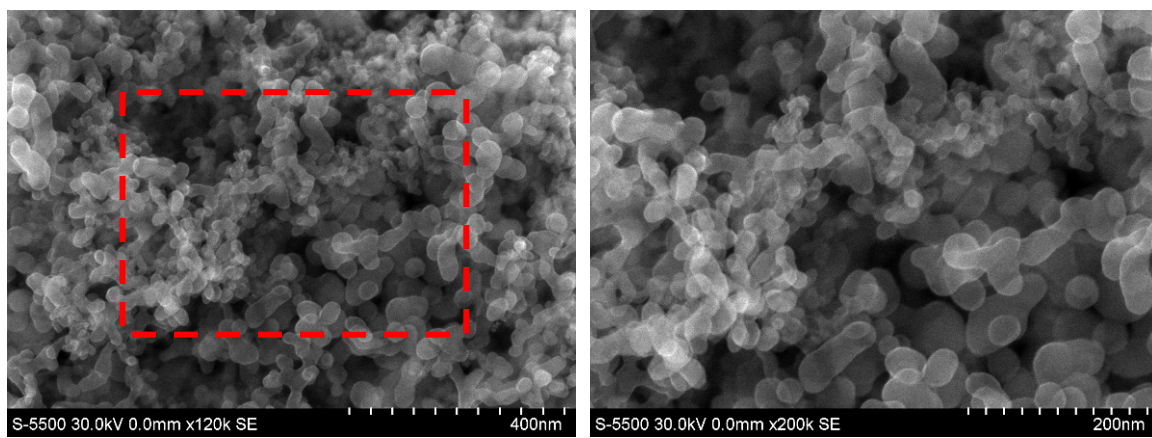


Figure 13. Secondary electron images of the silicon nanopowder.

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Should you have any questions regarding this information, please do not hesitate to contact us.

Sincerely,

A handwritten signature in cursive script, reading "Kristin L. Bunker".

Kristin L. Bunker, Ph.D.
Senior Scientist

cc: G. Casuccio (RJLG)



LABORATORY REPORT

Lawrence Berkeley Lab
One Cyclotron Rd.
MS 90J-01
Berkeley, CA 94720

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Phone: 510-486-7433
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Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q	
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)			
B3 - 16	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Aluminum	0.00700	70.0	0.00625	62.5	09/08/2009	DS
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Antimony	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Arsenic	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Barium	< 0.00250	< 25.0	0.00250	25.0	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Beryllium	< 0.00100	< 10.0	0.00100	10.0	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Bismuth	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Boron	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Cadmium	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Calcium	< 0.0125	< 125	0.0125	125	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Chromium	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Cobalt	< 0.00250	< 25.0	0.00250	25.0	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Copper	< 0.00375	< 37.5	0.00375	37.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Iron	0.0130	130	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Lead	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Lithium	4.93	49300	0.375	3750	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Magnesium	< 0.0125	< 125	0.0125	125	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Manganese	< 0.00250	< 25.0	0.00250	25.0	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Molybdenum	< 0.00250	< 25.0	0.00250	25.0	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Nickel	0.00128	12.8	0.00125	12.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Phosphorus	0.0130	130	0.00125	12.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Potassium	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Selenium	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Silicon	1.62	16200	0.250	2500	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Silver	< 0.00250	< 25.0	0.00250	25.0	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Sodium	0.0169	169	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Sulfur	< 0.0125	< 125	0.0125	125	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Thallium	< 0.00625	< 62.5	0.00625	62.5	09/08/2009	
	Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Tin	< 0.00250	< 25.0	0.00250	25.0	09/08/2009	
Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Titanium	46.0	460000	0.875	8750	09/08/2009		
Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Vanadium	< 0.00125	< 12.5	0.00125	12.5	09/08/2009		
Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Zinc	< 0.00625	< 62.5	0.00625	62.5	09/08/2009		
Lithium Titanate Nanopowder	10066036	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Zirconium	0.132	1320	0.00250	25.0	09/08/2009		



LABORATORY REPORT

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RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)		
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Aluminum	0.0191	191	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Antimony	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Arsenic	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Barium	< 0.00254	< 25.4	0.00254	25.4	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Beryllium	< 0.00102	< 10.2	0.00102	10.2	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Bismuth	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Boron	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Cadmium	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Calcium	< 0.0127	< 127	0.0127	127	09/08/2009	DS
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Chromium	0.0164	164	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Cobalt	< 0.0190	< 190	0.0190	190	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Copper	< 0.00381	< 38.1	0.00381	38.1	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Iron	31.7	317000	0.635	6350	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Lead	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Lithium	4.55	45500	0.0381	381	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Magnesium	< 0.0127	< 127	0.0127	127	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Manganese	0.161	1610	0.00254	25.4	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Molybdenum	< 0.00254	< 25.4	0.00254	25.4	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Nickel	0.00577	57.7	0.00127	12.7	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Phosphorus	17.7	177000	0.127	1270	09/08/2009	B
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Potassium	0.00656	65.6	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Selenium	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Silicon	0.0770	770	0.00254	25.4	09/08/2009	BS
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Silver	< 0.00254	< 25.4	0.00254	25.4	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Sodium	0.00849	84.9	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Sulfur	0.0631	631	0.0127	127	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Thallium	< 0.00635	< 63.5	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Tin	< 0.00254	< 25.4	0.00254	25.4	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Titanium	< 0.00888	< 88.8	0.00888	88.8	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Vanadium	< 0.00127	< 12.7	0.00127	12.7	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Zinc	0.00740	74.0	0.00635	63.5	09/08/2009	
Lithium Iron Phosphate Nanoparticles	10066037	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Zirconium	< 0.00254	< 25.4	0.00254	25.4	09/08/2009	



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RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q	
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)			
B3 - 18	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Aluminum	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	DS
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Antimony	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Arsenic	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Barium	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Beryllium	< 0.00102	< 10.2	0.00102	10.2	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Bismuth	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Boron	0.00682	68.2	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Cadmium	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Calcium	< 0.0128	< 128	0.0128	128	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Chromium	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Cobalt	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Copper	< 0.00383	< 38.3	0.00383	38.3	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Iron	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Lead	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Lithium	< 0.00383	< 38.3	0.00383	38.3	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Magnesium	< 0.0128	< 128	0.0128	128	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Manganese	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Molybdenum	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Nickel	< 0.00128	< 12.8	0.00128	12.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Phosphorus	0.00838	83.8	0.00128	12.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Potassium	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Selenium	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Silicon	21.9	219000	0.255	2550	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Silver	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Sodium	0.0106	106	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Sulfur	< 0.0128	< 128	0.0128	128	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Thallium	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Tin	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Titanium	< 0.00893	< 89.3	0.00893	89.3	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Vanadium	< 0.00128	< 12.8	0.00128	12.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Zinc	< 0.00638	< 63.8	0.00638	63.8	09/08/2009	
	Silicon Nanopowder	10066038	N/A	Vince Battaglia	Bldg. 70 Laboratory 295/299	Zirconium	< 0.00255	< 25.5	0.00255	25.5	09/08/2009	
											BS	



LABORATORY REPORT

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RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)		

Analyst Comments: ~20 mg of sample was placed in 50 mL centrifuge tube. 5 mL of nitric acid and 10 drops of hydrofluoric acid were added and they were digested for 1 hour at 100 °C. After that samples were cooled down, 3 mL of hydrochloric acid was added and samples were returned to hot block for another 30 minutes at 100 °C. Final volume was 25 mL. The digested samples were analyzed in a Varian 730ES ICP-OES (Inductive Coupled Plasma - Optical emission Spectroscopy).

Report Qualifiers (Q):

H = Holding times for preparation or analysis exceeded

P = NELAC[®] analyte certification pending

BS = Analyte not NELAC[®] certified

*NELAC-National Environmental Laboratory Accreditation Conference

E = Value above highest calibration standard

J = Value below lowest calibration standard but above MDL (Method Detection Limit)

L = LCS (Laboratory Control Standard)/SRM (Standard Reference Material) recovery outside accepted recovery limits

B = Analyte detected in the associated Method Blank

S = Spike Recovery outside accepted limits

R = RPD (relative percent difference) outside accepted limits

D = RL (reporting limit verification) outside accepted limits

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.

This laboratory operates in accord with ISO 17025:2005 guidelines, and holds a limited scope of accreditation under AIHA Lab ID 100364, NY ELAP Lab Code 10884, EPA Lab Code PA00162, CA ELAP Certificate 1970, PA DEP Lab ID 02-00396, VA DCLS Lab ID 00297, and LA DEQ Agency Interest 94775. This report may not be used to claim product endorsement by any laboratory accrediting agency. The results contained in this report relate only to the items tested or to the sample(s) as received by the laboratory. Any reproduction of this document must be in full for the report to be valid.

Quality Control data is available upon request. Results have not been blank corrected unless otherwise noted. Samples were received in good condition unless otherwise noted.



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

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RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID		Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
B3 - 20	PA-LRB:PAPB2-00909-03124-6300/QA1	Aluminum	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Antimony	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Arsenic	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Barium	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Beryllium	< 0.0080	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Bismuth	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Boron	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Cadmium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Calcium	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Chromium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Cobalt	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Copper	< 0.030	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Iron	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Lead	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Lithium	< 0.030	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Magnesium	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Manganese	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Molybdenum	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Nickel	< 0.010	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Phosphorus	0.055	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Potassium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Selenium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Silicon	1.864	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Silver	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Sodium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Sulfur	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Thallium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Tin	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Titanium	< 0.070	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Vanadium	< 0.010	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Zinc	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Zirconium	< 0.020	---	---	09/08/2009	



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

Lawrence Berkeley Lab
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MS 90J-01
Berkeley, CA 94720

Attn: Linnea Wahl
Phone: 510-486-7433
Fax: 510-486-6668
Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID	Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
PA-LFB:PAPB2-00909-03124-6300/QA2	Aluminum	0.93	1	93	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Antimony	0.92	1	92	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Arsenic	0.897	1	89.7	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Barium	0.913	1	91.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Beryllium	0.859	1	85.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Bismuth	0.914	1	91.4	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Boron	1.018	1	101.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Cadmium	0.868	1	86.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Calcium	0.832	1	83.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Chromium	0.925	1	92.5	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Cobalt	0.91	1	91	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Copper	0.951	1	95.1	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Iron	0.888	1	88.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Lead	0.893	1	89.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Lithium	0.863	1	86.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Magnesium	0.877	1	87.7	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Manganese	0.925	1	92.5	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Molybdenum	0.952	1	95.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Nickel	0.889	1	88.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Phosphorus	0.968	1	96.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Potassium	9.333	10	93.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Selenium	0.874	1	87.4	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Silicon	1.171	1	117.1	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Silver	0.889	1	88.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Sodium	0.936	1	93.6	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Sulfur	0.921	1	92.1	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Thallium	0.872	1	87.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Tin	0.856	1	85.6	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Titanium	0.952	1	95.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Vanadium	0.944	1	94.4	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Zinc	0.869	1	86.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Zirconium	0.948	1	94.8	09/08/2009	

B3 - 21



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Purchase Order No.: N/A

RJ Lee Group ID	Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
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Analyst Comments:

Report Qualifiers (Q):

H = Holding times for preparation or analysis exceeded

P = NELAC® analyte certification pending

N = Analyte not NELAC® certified

NELAC-National Environmental Laboratory Accreditation Conference

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This laboratory operates in accord with ISO 17025 guidelines, and holds limited scopes of accreditation under NY ELAP Lab Code 10884, EPA Lab Code PA00162, CA ELAP Certificate 1970, PA DEP Lab ID 02-00396, VA DCLS Lab ID 00297, and LA DEQ Agency Interest 94775.

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This report may not be used to claim product endorsement by any laboratory accrediting agency. The results contained in this report relate only to the items tested or to the sample(s) as received by the laboratory. Any reproduction of this document must be in full for the report to be valid. Quality Control data is available upon request.

E = Value above highest calibration standard but below LDR (Linear Dynamic Range)

J = Value below lowest calibration standard but above MDL (Method Detection Limit)

L = LCS (Laboratory Control Standard)/SRM (Standard Reference Material) recovery outside accepted recovery limits

B = Analyte detected in the associated Method Blank

S = Spike Recovery outside accepted recovery limits

R = RPD (relative percent difference) outside accepted recovery limits



September 18, 2009

Ms. Linnea Wahl
Pilot Study Project Manager
Ernest Orlando Lawrence Berkeley National Laboratory
Berkeley, CA 94720

Re: Electron Microscopy Results of Potential UNP
PI: Robert Kostecki: Building 70, Lab 108

Dear Linnea:

This report summarizes the electron microscopy (EM) results for the analysis of the samples collected from Building 70, Lab 108 as part of the Phase I pilot study related to potential unbound engineered nanoparticle (UNP) releases. Research in this laboratory is directed by Robert Kostecki and focuses on the optimization of the structure of carbon in lithium-ion composite electrodes intended for use in battery applications. The nanomaterial used in this research is lithium iron phosphate. In addition to battery applications, this laboratory has a graphene research program. Under Robert Kostecki's direction, Elad Pollak performs research related to graphene. Information related to the samples is listed in Table 1. The EM analysis was performed to provide information on particle size, morphological characteristics, and elemental composition associated with each material.

Table 1: Sample Information

Client Sample ID	RJLG Sample #	Sample Received Date	Sample Analysis Date	Analyst
Lithium Iron Phosphate	10066173	August 7, 2009	September 3, 2009	Kristin Bunker
Graphene	10066172	August 7, 2009	September 14, 2009	Brad Stitch

The lithium iron phosphate sample was characterized using an Hitachi S-5500 high resolution field emission scanning electron microscope (HR-FESEM) with scanning transmission electron microscopy (STEM) capabilities. The analysis was conducted primarily in the secondary and bright field transmission electron

modes at magnifications up to 1,000,000x. Compositional information was obtained through collection and processing of characteristic X-rays using a Bruker energy dispersive X-ray spectroscopy (EDS) system incorporating a 30 mm² silicon drift detector (SDD). The graphene sample collected on scotch tape was analyzed using an FEI Sirion 400 field emission SEM (FE-SEM) equipped with an Advanced Analysis Technologies EDS system with a silicon lithium detector. The analysis was conducted in the secondary electron mode at magnifications up to ~100,000x.

The lithium iron phosphate sample was analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES). A Varian 730ES ICP-OES was used to detect and quantify the elements in the material including trace metals by measuring light emitted at wavelengths characteristic of individual elements. The intensity of this emission is indicative of the concentration of the element within the sample.

The samples were prepared for ICP-OES using acid digestion techniques. The limited sample size and the unique composition of the samples did not allow for the preparation/digestion of any matrix duplicate (MD), matrix spike (MS) and laboratory control standard (LCS) quality control samples. A laboratory reagent blank (LRB) along with a fortified laboratory reagent blank (LFB) were prepared and analyzed instead. Elevated silicon (1.864 mg/L) in the LRB results was due to etching of the glass in the ICP-OES system by the hydrofluoric acid (HF) used to digest the samples. Elevated phosphorus (0.055 mg/L) in the LRB results was due to the sample container used to perform the digestion. All of the recoveries reported in the LFB results were within the acceptable laboratory limits ($\pm 15\%$), with the exception of silicon (for the same reasons as described above for the LRB) and calcium (which tends to form insoluble CaF_2 in the presence of HF). A detailed description of the sample preparation is described along with the results of the analysis in the table attached to this report.

The table below (Table 2) provides a concise summary of the EM analysis results. The sections following the table provide a summary of the EM results for the lithium iron phosphate and graphene.

Table 2: Sample Attributes

	Particle Size	Morphology	Chemistry
Lithium Iron Phosphate	Majority of particles were > 100 nm. Individual particles ranged from ~50 nm to 500 nm; Elongated particles often measured 100-200 nm in diameter and > 500 nm in length; Agglomerated structures on the order of micrometers	Elongated particles that were usually agglomerated	SEM/EDS: Fe, P and O; Trace amounts of Na, Cl, Si, K, S ICP-OES: Major elements included Fe, P, Li and trace amounts of Si, Mn, S, Al, Ca, Cr, Na, Zn
Graphene	Individual platelets were agglomerated and could not be distinguished, size information was difficult to determine; individual platelets were on the order of 200 nm	Slender platelets that appear to be agglomerated; unable to distinguish individual sheets	SEM/EDS: C

Lithium Iron Phosphate Sample

The lithium iron phosphate dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The EM analysis revealed that the sample was comprised primarily of individual rounded particles and elongated particles with rounded ends. The majority of the particles measured greater than 100 nanometers (nm), ranging from approximately 50-500 nm in average diameter. Elongated particles often measured 100-200 nm in diameter and greater than 500 nm in length. The particles had a smooth surface texture and were usually observed as agglomerates in the micrometer size range. EDS analysis indicated that the material was composed of iron (Fe), phosphorous (P), and oxygen (O), while lithium can not be detected using EDS analysis. EM images and an elemental spectrum are provided in Figures 1 and 2. Salt and carbon particles were also detected in trace amounts (see Figures 3 and 4).

The lithium iron phosphate sample was also analyzed by ICP-OES. The sample was comprised of Fe, P, lithium (Li) and trace amounts of silicon (Si), manganese

(Mn), sulfur (S), aluminum (Al), calcium (Ca), chromium (Cr), sodium (Na), and zinc (Zn). The results can be found at the end of the report.

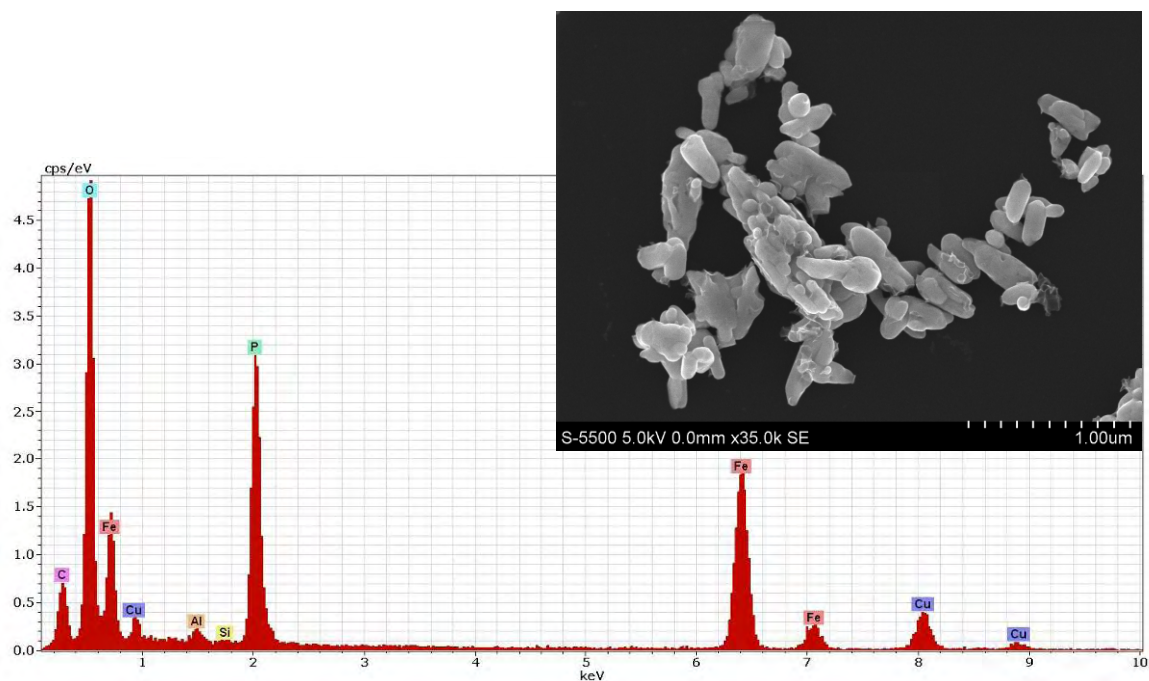


Figure 1. Secondary electron image along with an elemental spectrum of the lithium iron phosphate nanoparticles; (copper (Cu) X-rays are fluoresced from the TEM grid, carbon (C) from the substrate material, and aluminum (Al) from the sample holder).

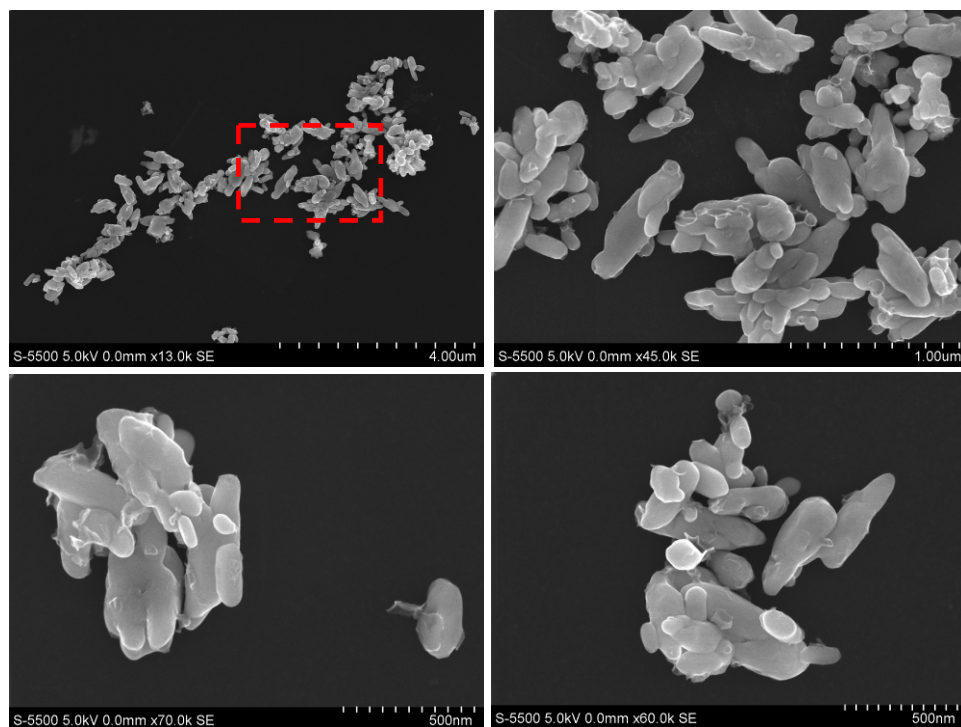


Figure 2. Secondary electron images of the lithium iron phosphate.

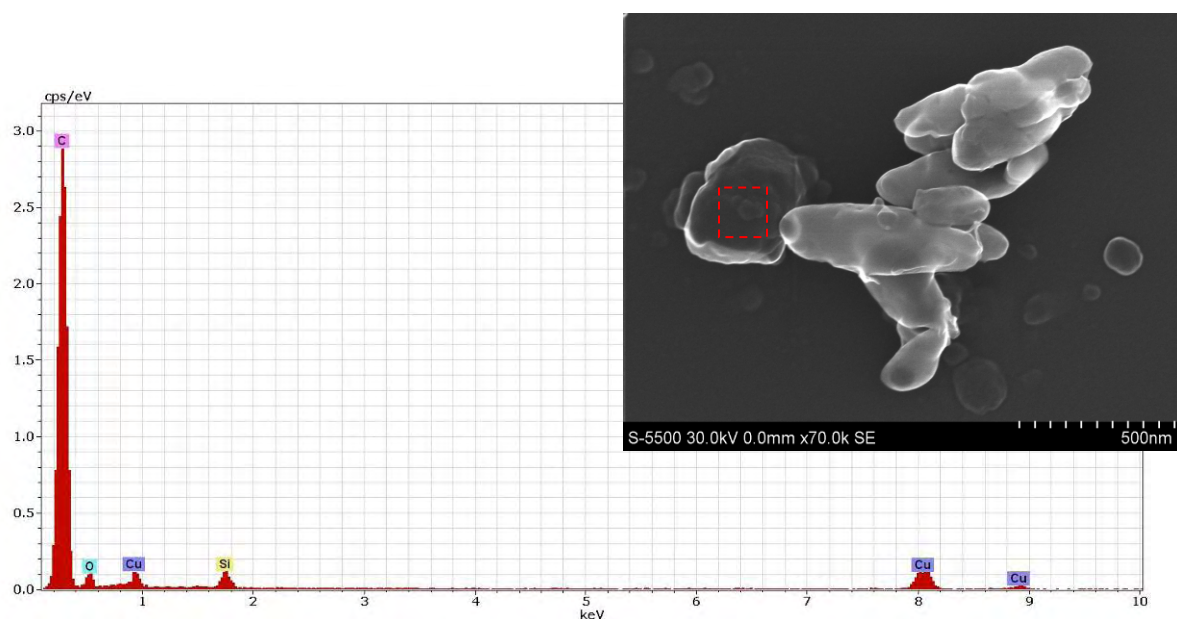


Figure 3. Secondary electron image along with an elemental spectrum of the carbon rich particles found on the sample.

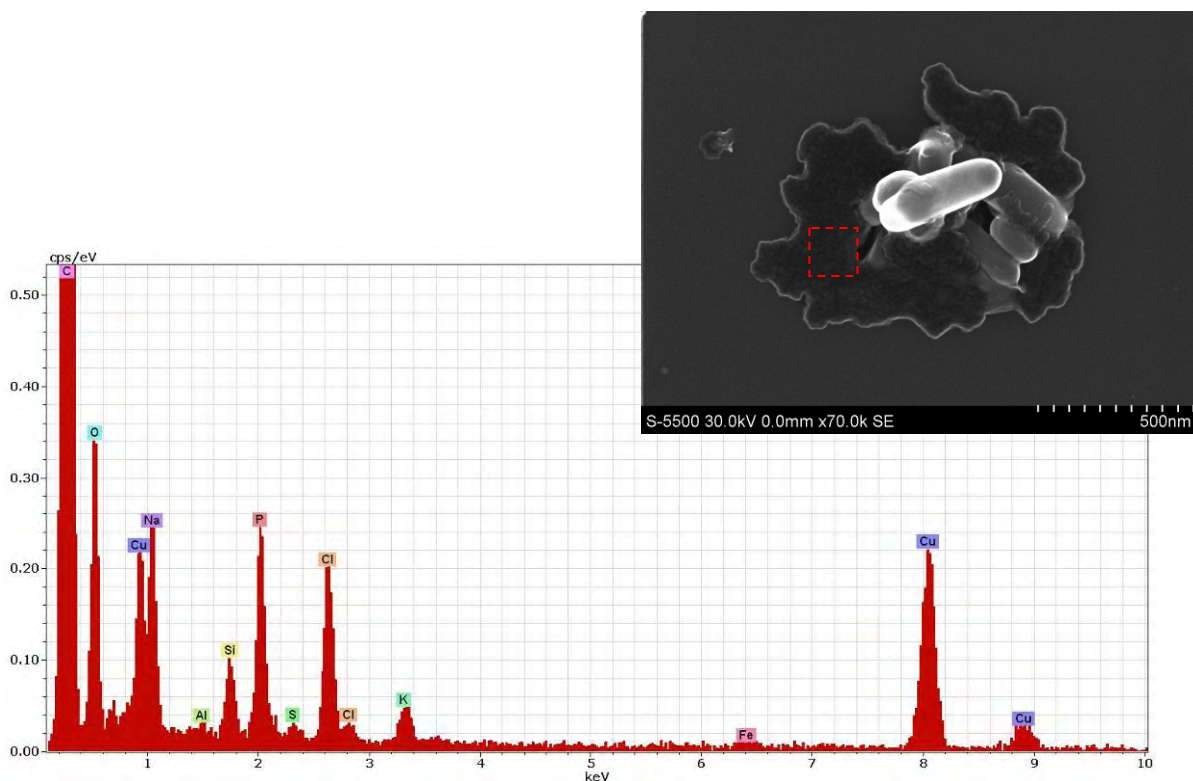


Figure 4. Secondary electron image along with an elemental spectrum of the salt material (Na-Cl with trace amounts of Si, K, and S) found on the sample.

Graphene

A portion of the graphene sample collected on scotch tape was mounted onto an SEM stub and coated with a thin layer of carbon by evaporative deposition to produce a conductive surface. The tape used to collect the sample was not well suited for SEM analysis.

Examination of the graphene sample in the FEI FE-SEM revealed that the sample was comprised of carbonaceous platelets. The size of the platelets appeared to range from approximately 200 nm to several micrometers in average diameter, although the material is assumed to be agglomerated and individual particle size was difficult to determine since individual sheets are difficult to distinguish. EM images and elemental spectra are provided in Figures 5 through 7.

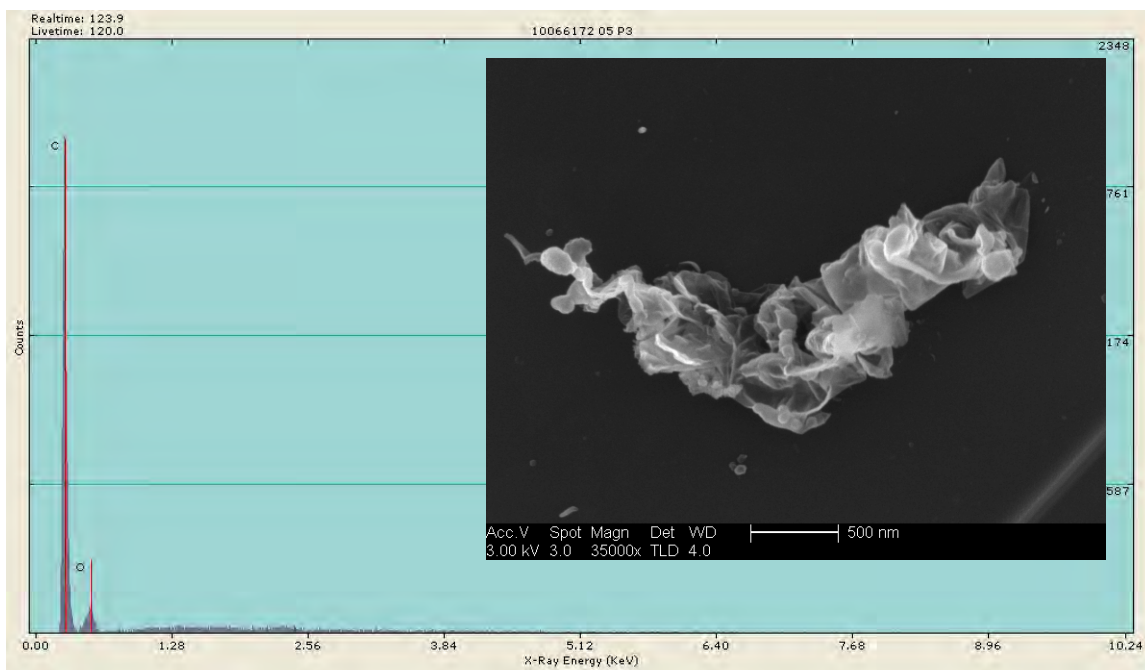


Figure 5. Secondary electron image and elemental spectrum of graphene.

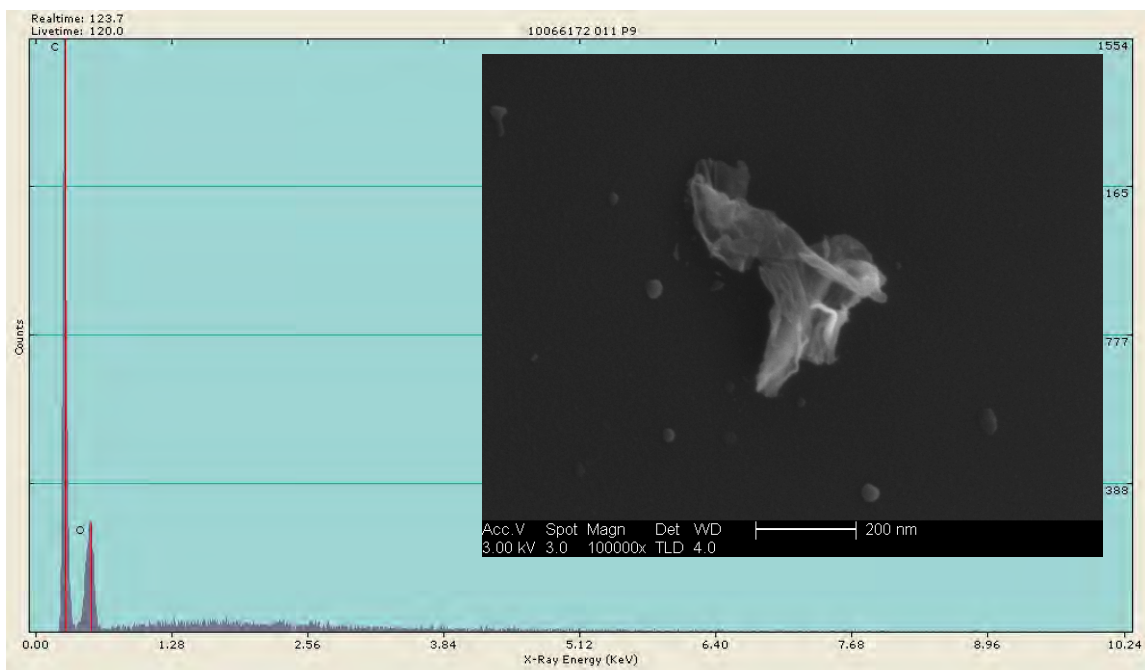


Figure 6. Secondary electron image and elemental spectrum of graphene.

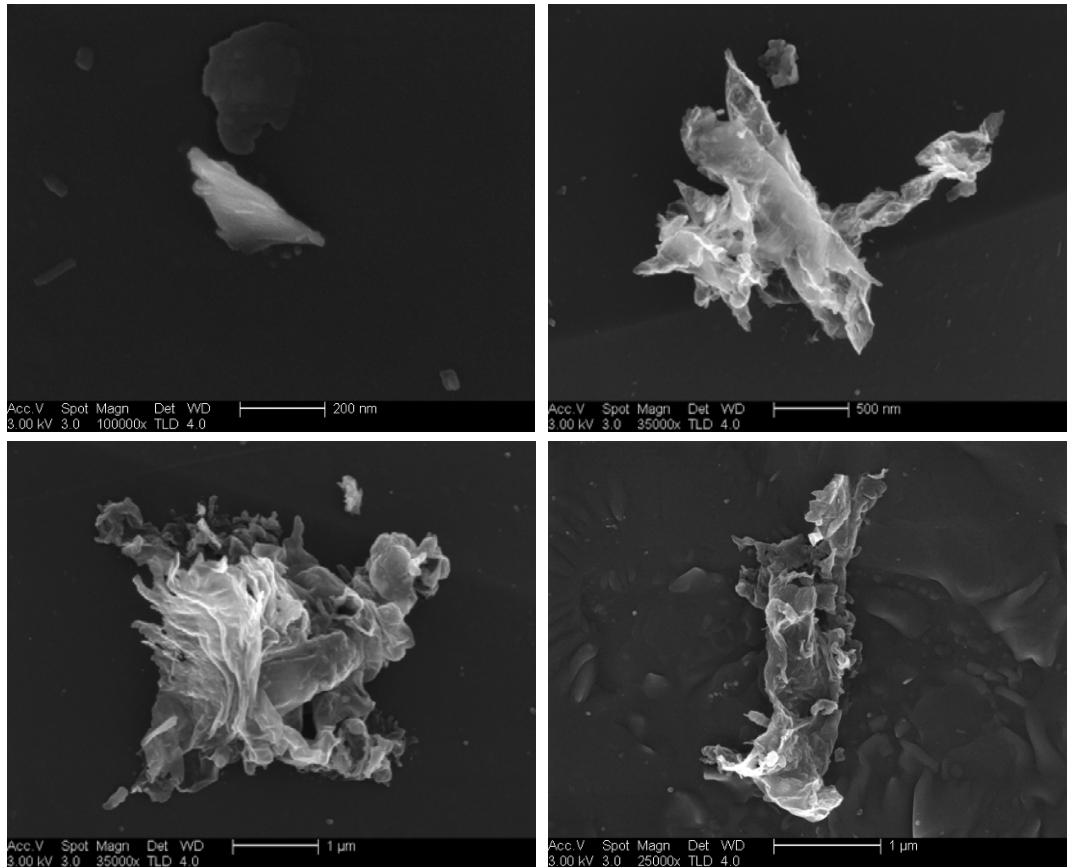


Figure 7. Secondary electron images of graphene.

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Should you have any questions regarding this information, please do not hesitate to contact us.

Sincerely,

Kristin L. Bunker, Ph.D.
Senior Scientist

cc: G. Casuccio (RJLG)



LABORATORY REPORT

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Berkeley, CA 94720

Attn: Linnea Wahl
Phone: 510-486-7433
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Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)		
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Aluminum	0.0172	172	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Antimony	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Arsenic	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Barium	< 0.00238	< 23.8	0.00238	23.8	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Beryllium	< 0.000952	< 9.52	0.000952	9.52	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Bismuth	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Boron	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Cadmium	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Calcium	0.0159	159	0.0119	119	09/08/2009	DS
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Chromium	0.0149	149	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Cobalt	< 0.0179	< 179	0.0179	179	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Copper	< 0.00357	< 35.7	0.00357	35.7	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Iron	32.1	321000	0.595	5950	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Lead	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Lithium	4.59	45900	0.0357	357	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Magnesium	< 0.0119	< 119	0.0119	119	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Manganese	0.167	1670	0.00238	23.8	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Molybdenum	< 0.00238	< 23.8	0.00238	23.8	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Nickel	0.00536	53.6	0.00119	11.9	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Phosphorus	17.9	179000	0.119	1190	09/08/2009	B
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Potassium	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Selenium	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Silicon	0.324	3240	0.0238	238	09/08/2009	BS
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Silver	< 0.00238	< 23.8	0.00238	23.8	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Sodium	0.0104	104	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Sulfur	0.0979	979	0.0119	119	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Thallium	< 0.00595	< 59.5	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Tin	< 0.00238	< 23.8	0.00238	23.8	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Titanium	< 0.00833	< 83.3	0.00833	83.3	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Vanadium	< 0.00119	< 11.9	0.00119	11.9	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Zinc	0.00610	61.0	0.00595	59.5	09/08/2009	
Lithium Iron Phosphate Nanomaterial	10066173	N/A	Robert Kostecki/Elad Pollak	Bldg. 70 Laboratory 108	Zirconium	< 0.00238	< 23.8	0.00238	23.8	09/08/2009	



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Report Date: September 14, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A
Matrix: Solid
Prep/Analysis: EPA 3050B / EPA 6010C (No LCS Solid)-PA

Client Sample ID	RJ Lee Group ID	Sampling Date	Researcher	Site	Analyte	Sample Concentration		Minimum Reporting Limit		Analysis Date	Q
						Weight Percent (%)	Parts per Million (PPM)	Weight Percent (%)	Parts per Million (PPM)		
Analyst Comments: ~20 mg of sample was placed in 50 mL centrifuge tube. 5 mL of nitric acid and 10 drops of hydrofluoric acid were added and they were digested for 1 hour at 100 °C. After that samples were cooled down, 3 mL of hydrochloric acid was added and samples were returned to hot block for another 30 minutes at 100 °C. Final volume was 25 mL. The digested samples were analyzed in a Varian 730ES ICP-OES (Inductive Coupled Plasma - Optical emission Spectroscopy).											

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*NELAC-National Environmental Laboratory Accreditation Conference

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Quality Control data is available upon request. Results have not been blank corrected unless otherwise noted. Samples were received in good condition unless otherwise noted.

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J = Value below lowest calibration standard but above MDL (Method Detection Limit)

L = LCS (Laboratory Control Standard)/SRM (Standard Reference Material) recovery

outside accepted recovery limits

B = Analyte detected in the associated Method Blank

S = Spike Recovery outside accepted limits

R = RPD (relative percent difference) outside accepted limits

D = RL (reporting limit verification) outside accepted limits



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

Lawrence Berkeley Lab
One Cyclotron Rd.
MS 90J-01
Berkeley, CA 94720

Attn: Linnea Wahl
Phone: 510-486-7433
Fax: 510-486-6668
Email: lewahl@lbl.gov

RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID		Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
B4 - 11	PA-LRB:PAPB2-00909-03124-6300/QA1	Aluminum	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Antimony	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Arsenic	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Barium	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Beryllium	< 0.0080	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Bismuth	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Boron	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Cadmium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Calcium	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Chromium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Cobalt	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Copper	< 0.030	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Iron	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Lead	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Lithium	< 0.030	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Magnesium	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Manganese	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Molybdenum	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Nickel	< 0.010	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Phosphorus	0.055	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Potassium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Selenium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Silicon	1.864	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Silver	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Sodium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Sulfur	< 0.10	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Thallium	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Tin	< 0.020	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Titanium	< 0.070	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Vanadium	< 0.010	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Zinc	< 0.050	---	---	09/08/2009	
	PA-LRB:PAPB2-00909-03124-6300/QA1	Zirconium	< 0.020	---	---	09/08/2009	



LABORATORY REPORT-PREPARATION QA/QC SUMMARY

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Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID	Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
PA-LFB:PAPB2-00909-03124-6300/QA2	Aluminum	0.93	1	93	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Antimony	0.92	1	92	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Arsenic	0.897	1	89.7	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Barium	0.913	1	91.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Beryllium	0.859	1	85.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Bismuth	0.914	1	91.4	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Boron	1.018	1	101.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Cadmium	0.868	1	86.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Calcium	0.832	1	83.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Chromium	0.925	1	92.5	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Cobalt	0.91	1	91	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Copper	0.951	1	95.1	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Iron	0.888	1	88.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Lead	0.893	1	89.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Lithium	0.863	1	86.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Magnesium	0.877	1	87.7	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Manganese	0.925	1	92.5	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Molybdenum	0.952	1	95.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Nickel	0.889	1	88.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Phosphorus	0.968	1	96.8	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Potassium	9.333	10	93.3	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Selenium	0.874	1	87.4	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Silicon	1.171	1	117.1	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Silver	0.889	1	88.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Sodium	0.936	1	93.6	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Sulfur	0.921	1	92.1	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Thallium	0.872	1	87.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Tin	0.856	1	85.6	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Titanium	0.952	1	95.2	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Vanadium	0.944	1	94.4	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Zinc	0.869	1	86.9	09/08/2009	
PA-LFB:PAPB2-00909-03124-6300/QA2	Zirconium	0.948	1	94.8	09/08/2009	

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LABORATORY REPORT-PREPARATION QA/QC SUMMARY

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RJ Lee Group Job No.: GSG905991
RJ Lee Group Chemistry Job No.: IN02092009P009
Samples Received: September 2, 2009
Report Date: September 24, 2009
Client Project: Environ Assessment at LBNL
Purchase Order No.: N/A

RJ Lee Group ID	Analyte	Instrument result (mg/L)	Nominal Value (mg/L)	Recovery (%)	Analysis Date	Q
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Analyst Comments:

Report Qualifiers (Q):
H = Holding times for preparation or analysis exceeded
P = NELAC® analyte certification pending
N = Analyte not NELAC® certified
NELAC-National Environmental Laboratory Accreditation Conference
These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.
This laboratory operates in accord with ISO 17025 guidelines, and holds limited scopes of accreditation under NY ELAP Lab Code 10884, EPA Lab Code PA00162, CA ELAP Certificate 1970, PA DEP Lab ID 02-00396, VA DCLS Lab ID 00297, and LA DEQ Agency Interest 94775.
This report may not be used to claim product endorsement by any laboratory accrediting agency. The results contained in this report relate only to the items tested or to the sample(s) as received by the laboratory. Any reproduction of this document must be in full for the report to be valid. Quality Control data is available upon request.

E = Value above highest calibration standard but below LDR (Linear Dynamic Range)
J = Value below lowest calibration standard but above MDL (Method Detection Limit)
L = LCS (Laboratory Control Standard)/SRM (Standard Reference Material) recovery outside accepted recovery limits
B = Analyte detected in the associated Method Blank
S = Spike Recovery outside accepted recovery limits
R = RPD (relative percent difference) outside accepted recovery limits



September 18, 2009

Ms. Linnea Wahl
Pilot Study Project Manager
Ernest Orlando Lawrence Berkeley National Laboratory
Berkeley, CA 94720

Re: Electron Microscopy Results of Potential UNP
PI: Rick Russo: Building 70, Lab 157

Dear Linnea:

This report summarizes the electron microscopy (EM) results from the analysis of the sample collected from Building 70, Lab 157 as part of the Phase I pilot study related to potential unbound engineered nanoparticle (UNP) releases. Research conducted in this laboratory is directed by Rick Russo and performed by Xianglei Mao. The research focuses on fundamental studies related to laser ablation and the nanomaterial used in this research is iron disulfide. Information related to the sample is listed in Table 1. The EM analysis was performed to provide information on particle size, morphological characteristics, and elemental composition associated with the material.

Table 1: Sample Information

Client Sample ID	RJLG Sample #	Sample Received Date	Sample Analysis Date	Analyst
Iron Disulfide	10066174	August 7, 2009	September 2, 2009	Traci Lersch

The sample was characterized using an Hitachi S-5500 high resolution field emission scanning electron microscope (HR-FESEM) with scanning transmission electron microscopy (STEM) capabilities. The analysis was conducted primarily in the secondary and bright field transmission electron modes at magnifications up to 1,000,000x. Compositional information was obtained through collection and processing of characteristic X-rays using a Bruker energy dispersive X-ray spectroscopy (EDS) system incorporating a 30 mm² silicon drift detector (SDD).

The table below (Table 2) provides a concise summary of the EM analysis results. The section following the table provides a summary of the EM results for the iron disulfide sample.

Table 2: Sample Attributes

	Particle Size	Morphology	Chemistry
Iron Disulfide	Droplets measured ~20 to 80 nm; Agglomerated structures on the order of micrometers	Majority of the particles by number consisted of Fe-S in the form of a droplet. Agglomerated Fe-S particles on the order of micrometers also present	SEM/EDS: Major: Fe-S; Minor: Ca-Fe-S; Trace: Si-rich

Iron Disulfide Sample

The iron disulfide dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The analysis revealed that iron-sulfur (Fe-S) droplets measuring ~20 to 80 nanometers (nm) were dominant by number. Droplets appear to have deposited on the filter in a liquid form. A minor amount of particles in the 20-80 nm size range consisted of Ca-S-Fe and a trace of silicate particles were also observed from ~50 nm to 5 micrometers in average diameter. EM images and elemental spectra are provided in Figures 1 and 2. Fe-S droplets appear to collect to form a large mass of the material in areas (see Figure 3). Agglomerated Fe-S particles on the order of micrometers with a rough surface texture accounted for a significant concentration of the sample mass (see Figure 4). EM of a silicate particle measuring greater than 5 μm is illustrated in Figure 5.

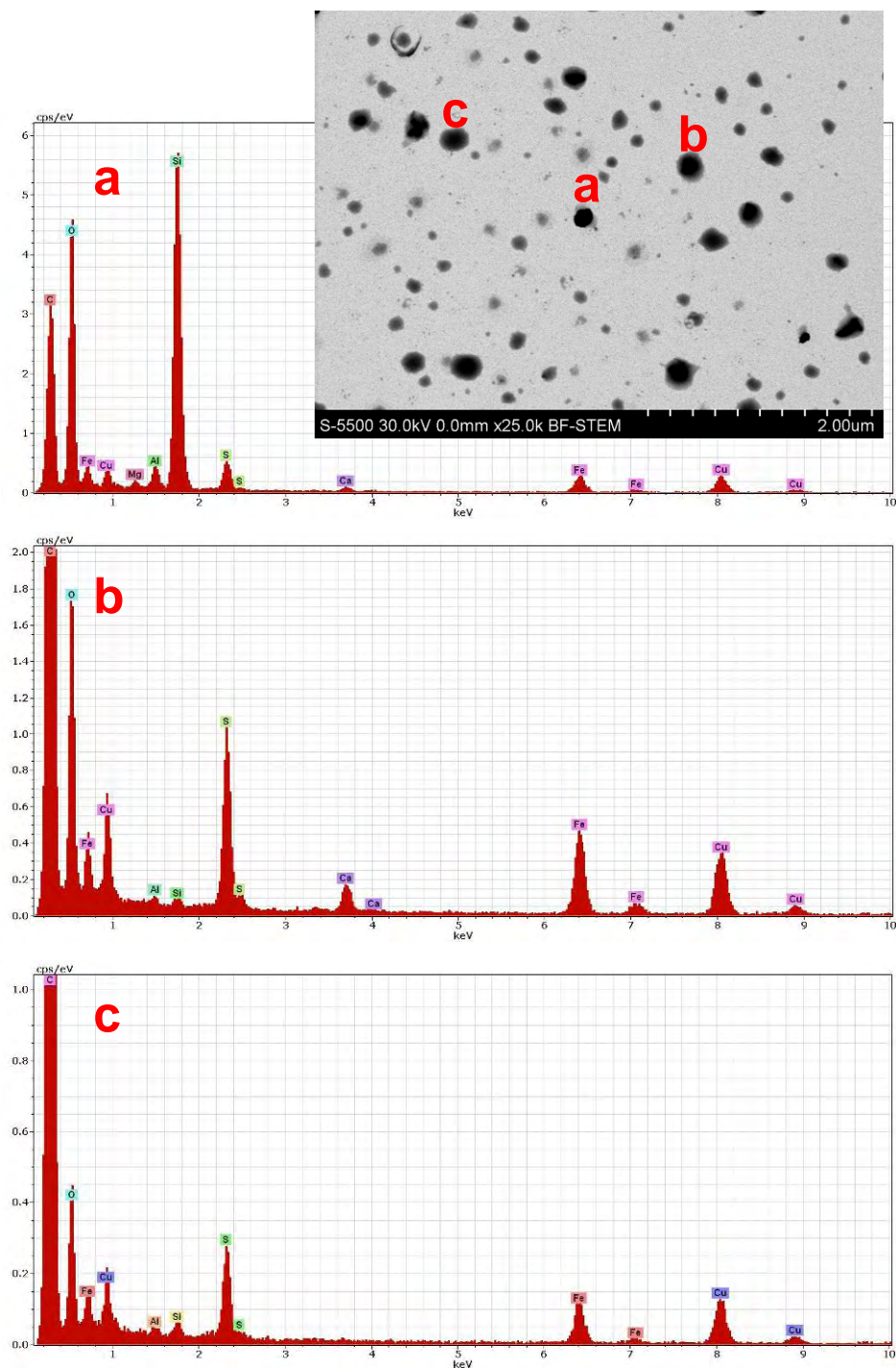


Figure 1. Bright field STEM image and elemental spectra of the iron disulfide sample. The sample consisted of droplets comprised of Fe-S, Ca-Fe-S and Si-rich particles. (Copper (Cu) X-rays are fluoresced from the TEM grid, carbon (C) from the substrate material, and aluminum (Al) from the sample holder).

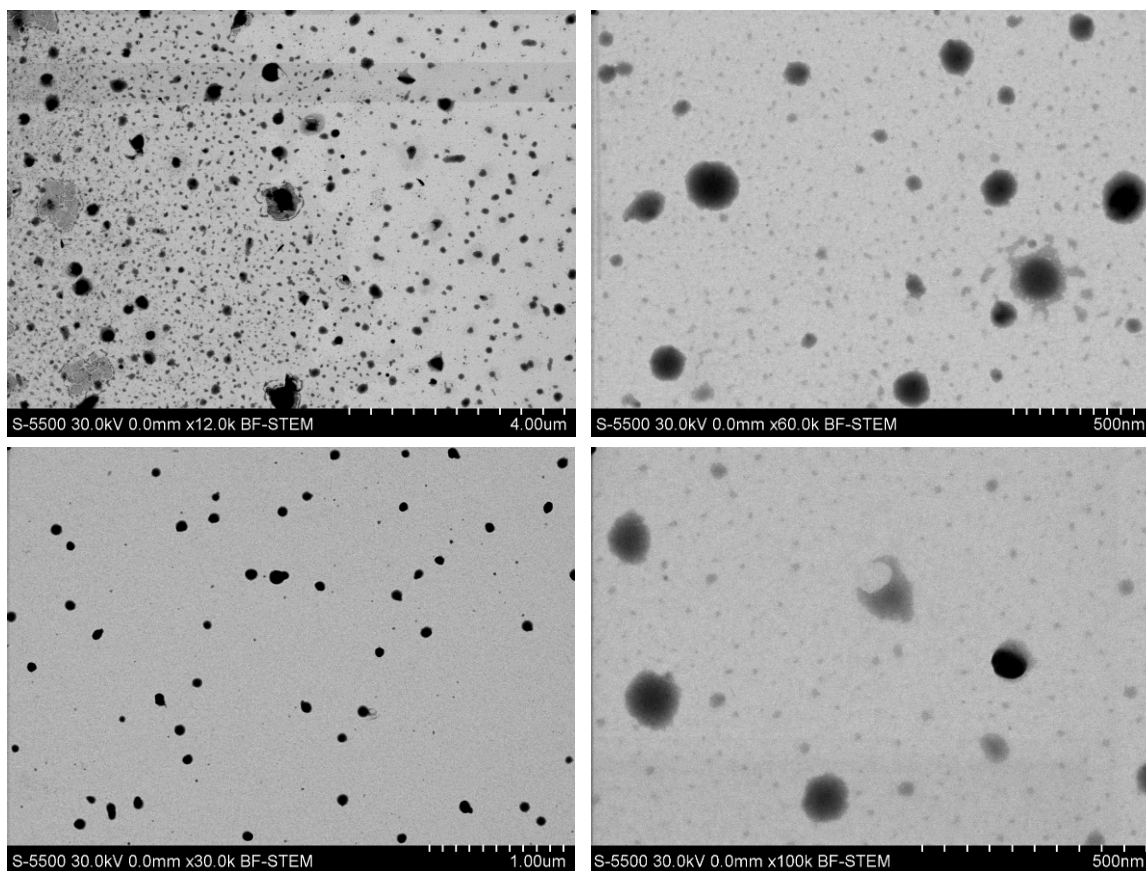


Figure 2. Bright field STEM images of the iron disulfide sample displayed at variable magnifications consisting primarily of Fe-S droplets.

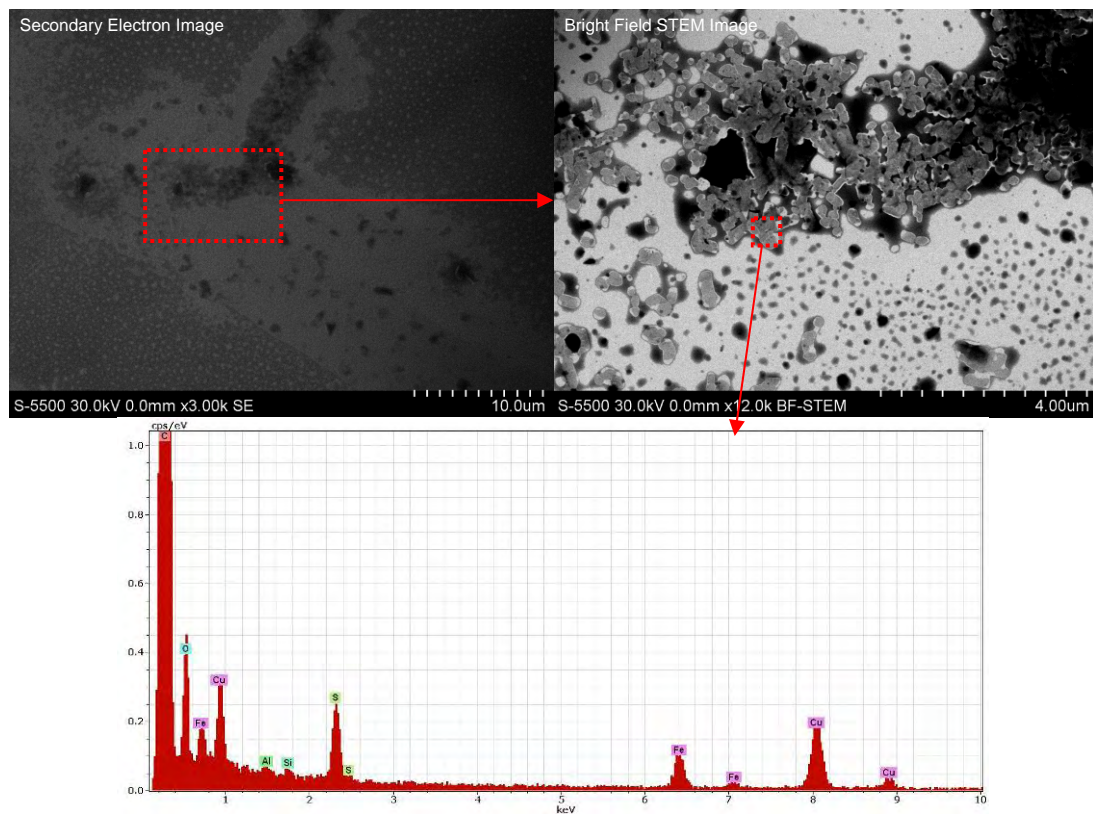


Figure 3. Secondary electron and bright field STEM images of the iron disulfide sample.

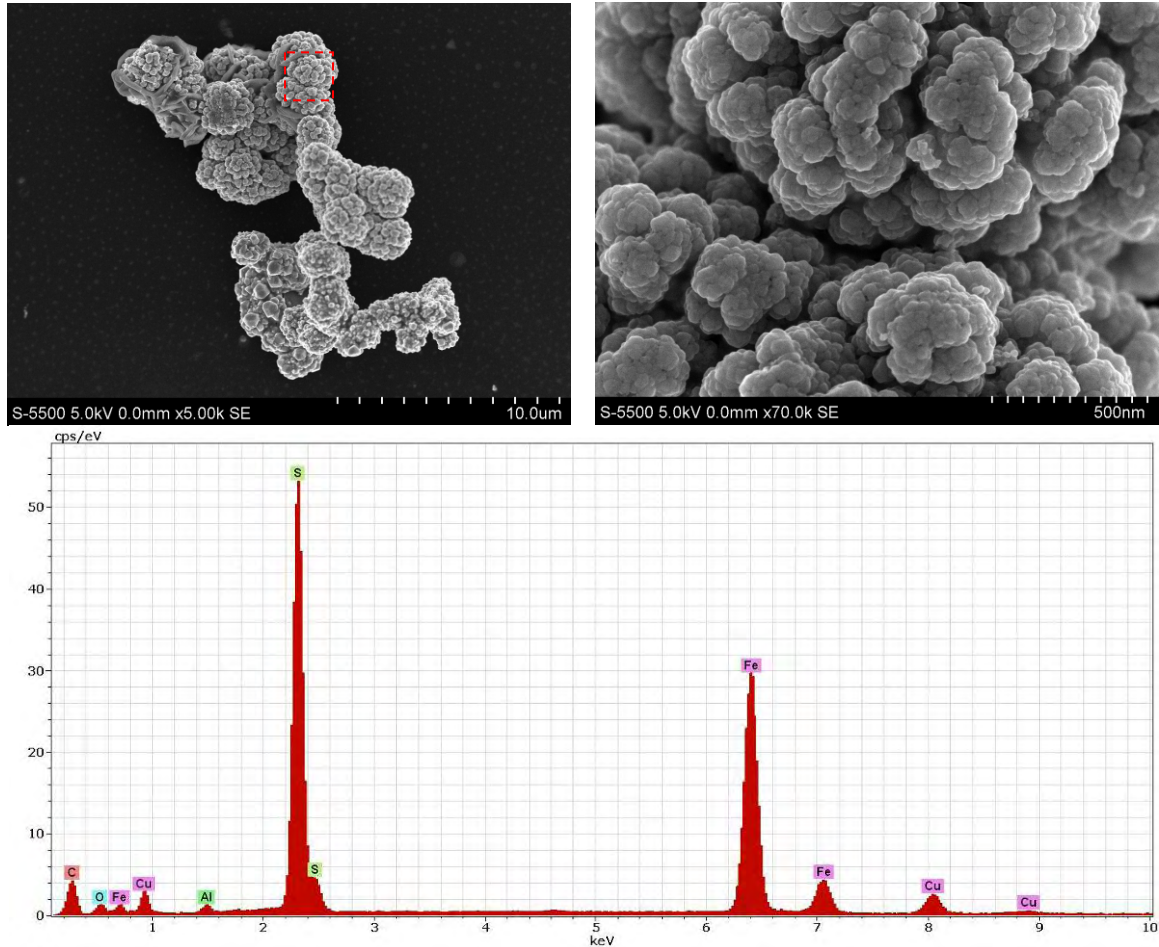


Figure 4. Secondary electron image along with an elemental spectrum of iron disulfide particles.

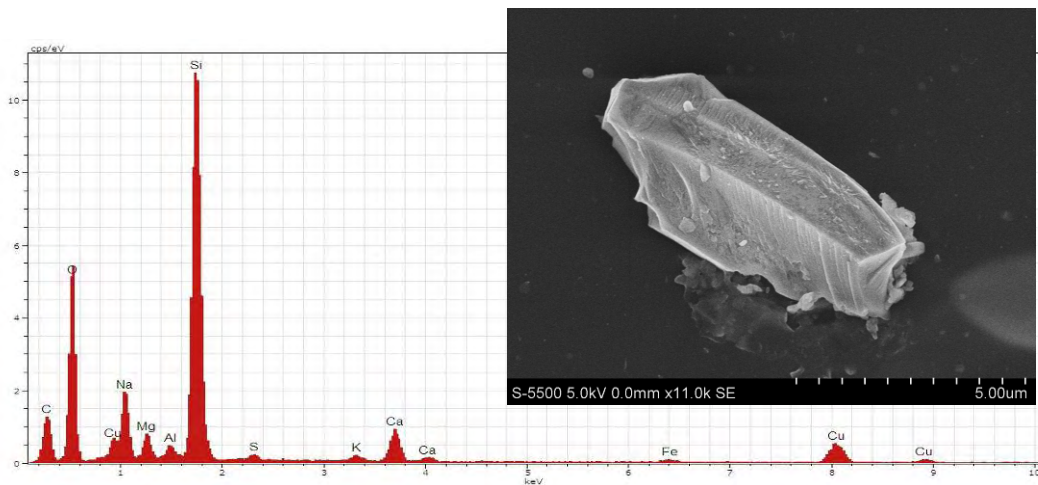
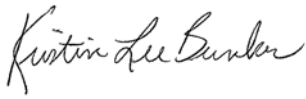


Figure 5. Secondary electron image along with an elemental spectrum of a silicon-rich particle.

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted.

Should you have any questions regarding this information, please do not hesitate to contact us.

Sincerely,

A handwritten signature in cursive script, reading "Kristin L. Bunker".

Kristin L. Bunker, Ph.D.
Senior Scientist

cc: G. Casuccio (RJLG)



September 18, 2009

Ms. Linnea Wahl
Pilot Study Project Manager
Ernest Orlando Lawrence Berkeley National Laboratory
Berkeley, CA 94720

Re: Electron Microscopy Results of Potential UNP
PI: Don Lucas: Building 70, Lab 291/293

Dear Linnea:

This report summarizes the electron microscopy (EM) results for the analysis of the samples collected from Building 70, Lab 291/293 as part of the Phase I pilot study related to potential unbound engineered nanoparticle (UNP) releases. Research conducted in this laboratory is directed by Don Lucas and focuses on methods to detect toxic species using optical sensors. Nanomaterials being utilized in these research areas include gold nanorods, gold nanospheres, and oxidized soot. Information related to the samples is listed in Table 1. The EM analysis was performed to provide information on particle size, morphological characteristics, and elemental composition associated with each material.

Table 1: Sample Information

Client Sample ID	RJLG Sample #	Sample Received Date	Sample Analysis Date	Analyst
Au Nanorods	10066021	August 5, 2009	September 1, 2009	Kristin Bunker
Au Nanospheres	10066022	August 5, 2009	September 1, 2009	Kristin Bunker
Oxidized Soot	10066023	August 5, 2009	September 1, 2009	Kristin Bunker

The samples were characterized using an Hitachi S-5500 high resolution field emission scanning electron microscope (HR-FESEM) with scanning transmission electron microscopy (STEM) capabilities. The analysis was conducted primarily in the secondary and bright field transmission electron modes at magnifications up to 1,000,000x. Compositional information was obtained through collection

and processing of characteristic X-rays using a Bruker energy dispersive X-ray spectroscopy (EDS) system incorporating a 30 mm² silicon drift detector (SDD).

The table below (Table 2) provides a concise summary of the EM analysis results. The sections following the table provide a summary of the EM results for the gold nanorods, gold nanospheres, and oxidized soot samples.

Table 2: Sample Attributes

	Particle Size	Morphology	Chemistry
Au Nanorods	Rod-shaped particles ~20 nanometers (nm) in diameter and ~50 nm in length; Rounded and spherical particles were ~40-50 nm in diameter	Primarily rod-shaped particles; Rounded and spherical particles; observed in clusters	SEM/EDS: Au with Si residue
Au Nanospheres	Sizes ranged from ~3 to 125 nm. Agglomerates as large as several micrometers	Irregularly shaped and angular particles were predominant in the larger size fraction. Particles <~50 nm were spherical. A trace of rod shaped particles were detected	SEM/EDS: Au with Si residue
Oxidized Soot	Primary particles ~20-40 nm; Agglomerated structures on the order of micrometers	Aciniform structures comprised of rounded and irregularly shaped particles	SEM/EDS: C

Gold Nanorods

An aqueous solution (~ 5 ml) containing gold nanoparticles identified as nanorods was provided for EM analysis. Sample preparation involved dispersing the particles in the solution using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The EM analysis revealed that the majority of the sample was comprised of rod-shaped particles with rounded ends averaging 20 nanometers (nm) in diameter and 50 nm in length. EDS analysis confirmed that the material was composed of gold (Au). An EM micrograph and elemental spectrum of gold nanorods are presented in Figure 1. Rounded and spherical particles were also observed averaging 40-50 nm in diameter. Both the rod-shaped and rounded particles appeared to have a smooth surface texture. Individual particles in the EM image

provided in Figure 2 were measured using an image analysis program (i.e., analySIS¹) and the size dimensions are overlaid on the image. Particles had a tendency to collect in mono-layered clusters aligned in an ordered orientation. This is further illustrated in the additional images provided in Figure 3. The majority of the Au nanoparticles can be found encompassed in a residual material. The dendrite formation of this material surrounding the particles is shown in the low magnification bright and dark field images provided in Figure 4. EDS analysis of the residue surrounding the particles was comprised of carbon and a trace amount of silicon.

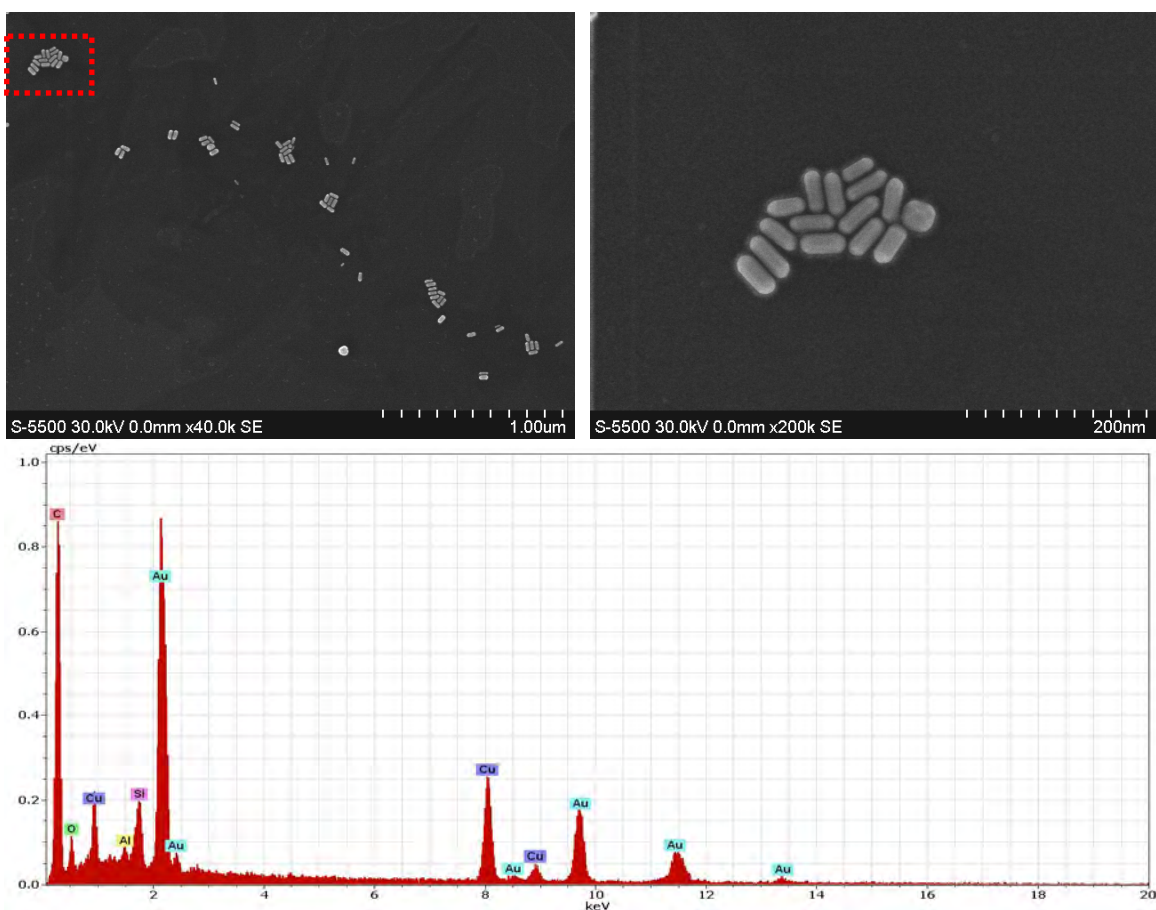


Figure 1. Secondary electron images and elemental spectrum of gold nanorods; (copper (Cu) X-rays are fluoresced from the TEM grid, carbon (C), oxygen (O), and aluminum (Al) from the sample holder).

¹ analySIS®; Digital Solutions for Imaging and Microscopy Soft Imaging System; www.soft-imaging.net

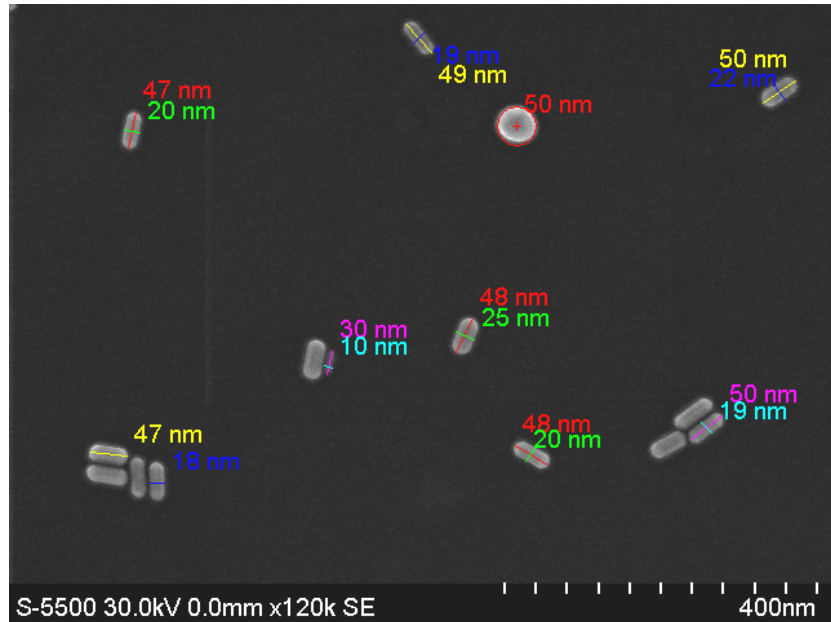


Figure 2. Secondary image illustrating size dimensions of individual particles.

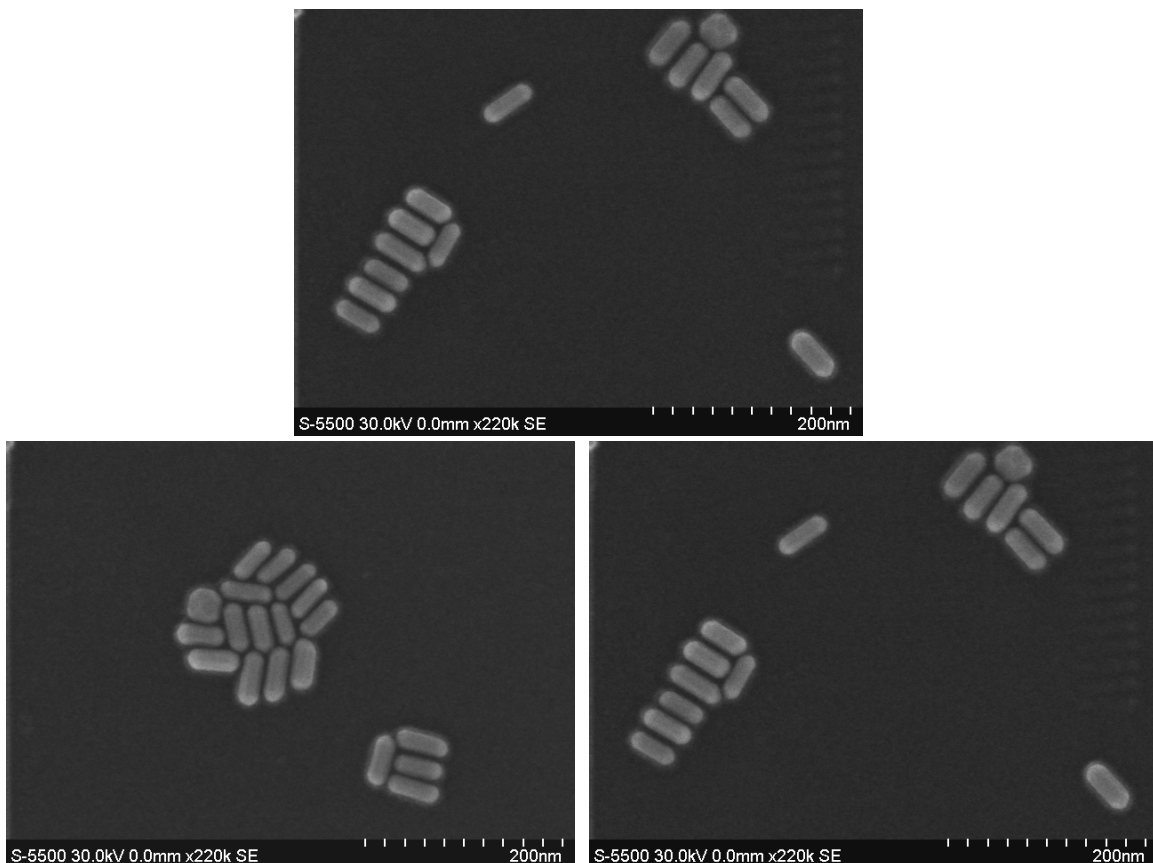


Figure 3. Secondary electron images of mono-layered clusters of gold nanorods.

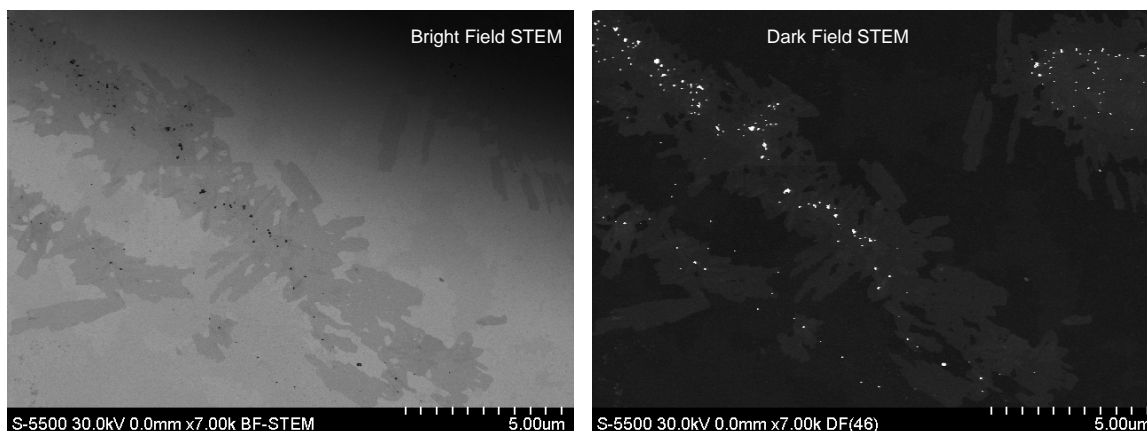


Figure 4. Dendrite formation of a residual material surrounding the Au particles shown in the low magnification bright and dark field images.

Gold Nanospheres

An aqueous solution (~ 20 ml) containing silver nanoparticles was provided for EM analysis. Sample preparation involved dispersing the particles in the solution using ultrasonic agitation. A carbon substrate supported by a transmission electron microscope (TEM) grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The EM analysis revealed a tri-modal distribution of gold particles on the sample ranging from approximately 3 nm to 125 nm in average diameter. The EM micrographs presented in Figure 5 show the variable size distribution. Irregularly shaped and angular particles are predominant in the larger size fraction, whereas particles measuring less than ~50 nm are typically spherical. Ultra-fine spherical particles observed only in transmission mode measuring 3-10 nm were dominant by number. Particles greater than approximately 10 nm were primarily agglomerated on the order of micrometers; whereas 3-10 nm particles were clustered without contact (see Figure 6). Individual particles were measured using the analySIS program and the EM images along with size dimension overlays are provided in Figure 7. Traces of rod shaped particles with rounded ends ~20 nm in diameter were also observed. The EDS analysis of the material confirmed that the particles were composed of gold (Au). Figures 8 and 9 provide images with elemental spectra. Sodium-sulfur (Na-S) material was associated with the sample as shown in Figures 10 and 11. There was a residue associated with the particles on the sample. EDS analysis of the residue surrounding the particles was comprised of carbon and a trace amount of silicon.

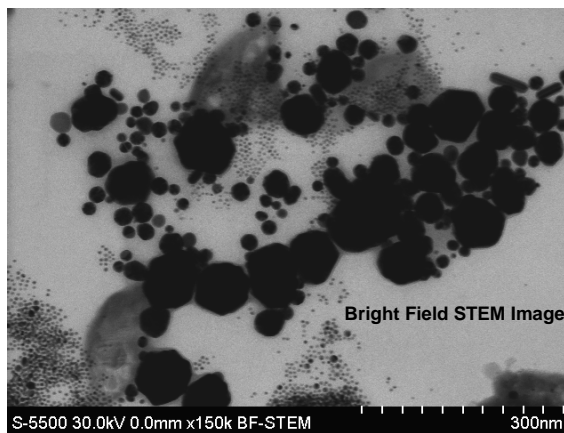


Figure 5. Secondary electron and bright field STEM images of gold nanospheres illustrating the variability in particle size.

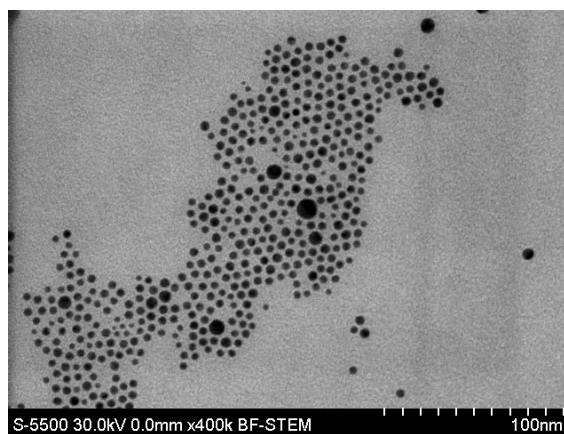


Figure 6. Bright Field STEM image of ultra-fine spherical particles observed only in transmission mode measuring approximately 3 to 10 nm.

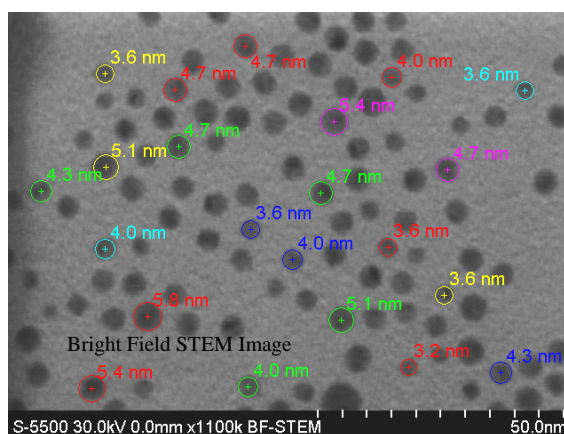


Figure 7. Secondary electron and bright field STEM images of the gold nanosphere sample with size dimensions.

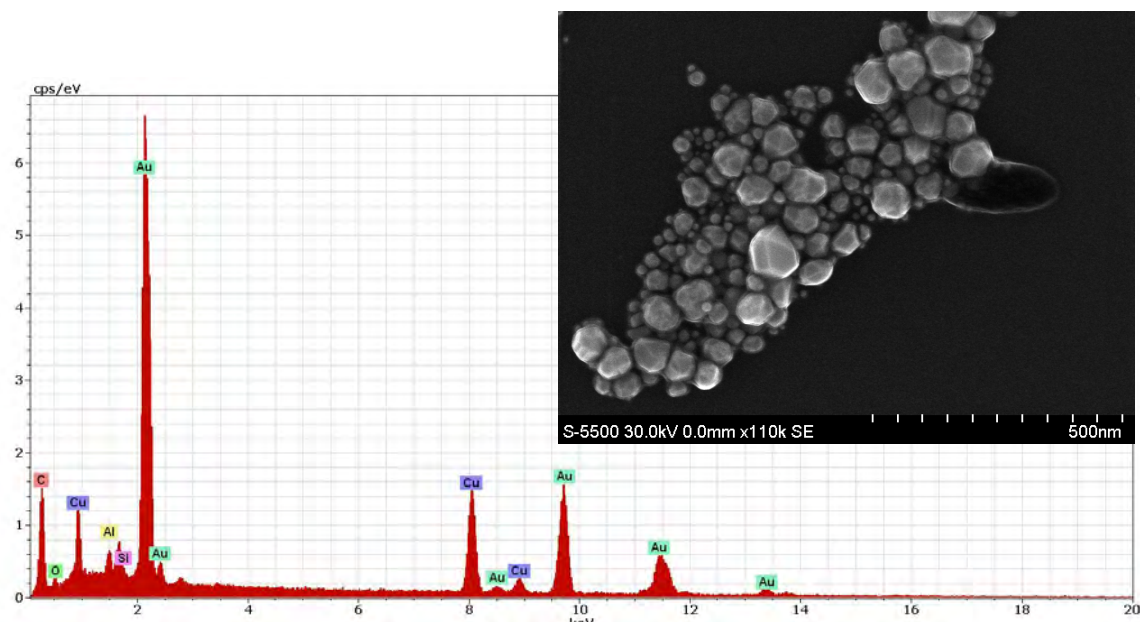


Figure 8. Secondary electron image and elemental spectrum of gold nanospheres.

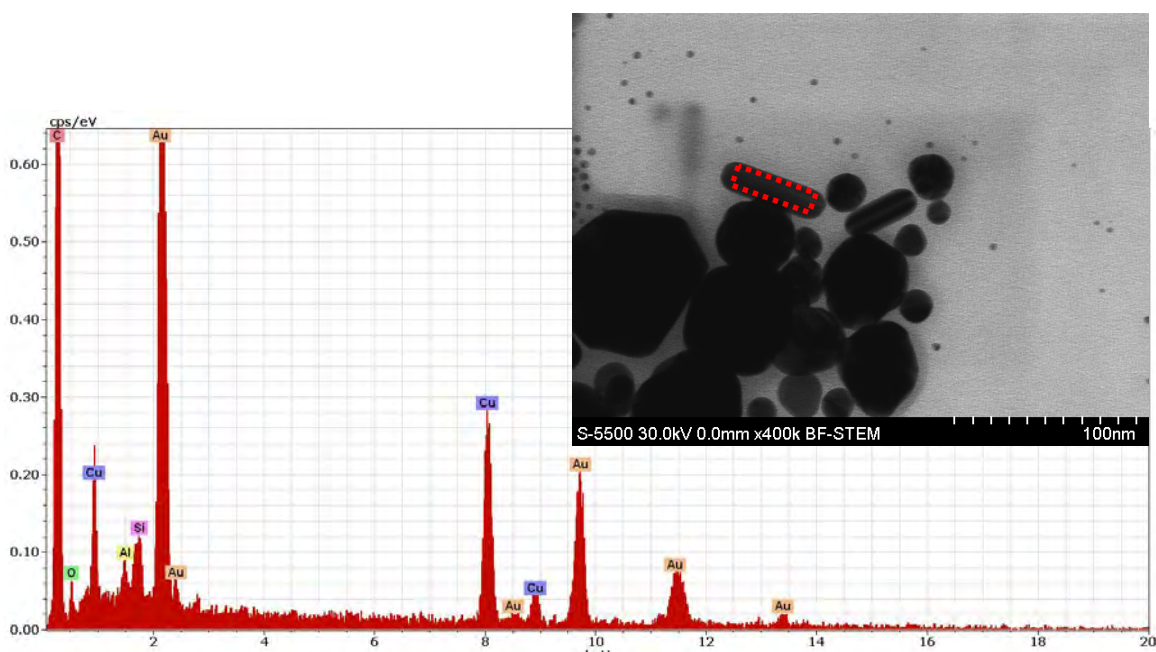


Figure 9. Bright field STEM image and elemental spectrum of rod-shaped particles on the gold nanosphere sample.

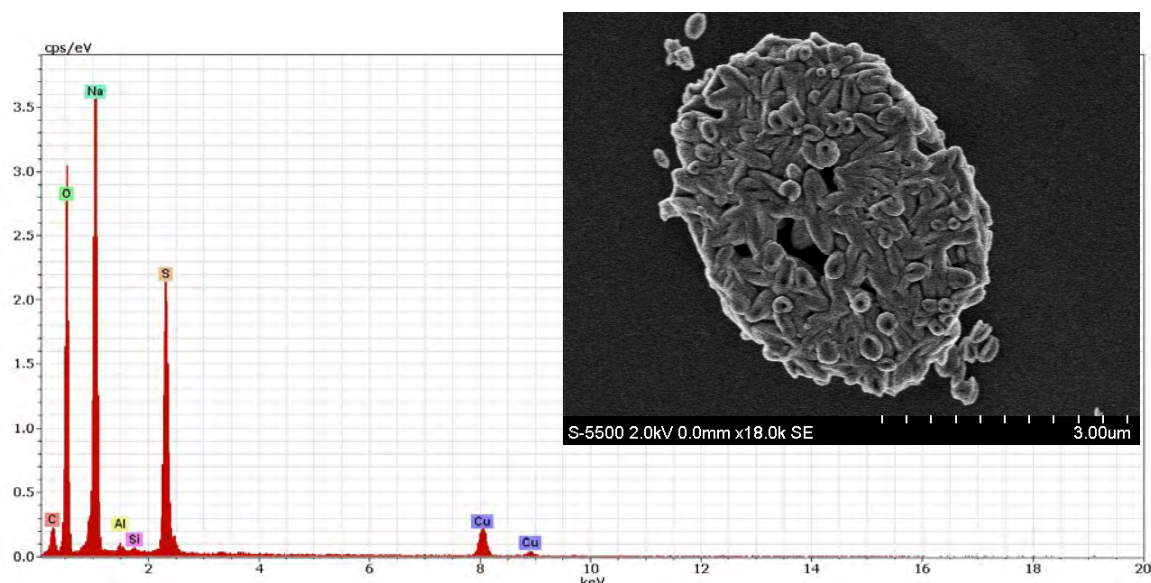


Figure 10. Secondary electron image and elemental spectrum of Na-S material on the gold nanosphere sample.

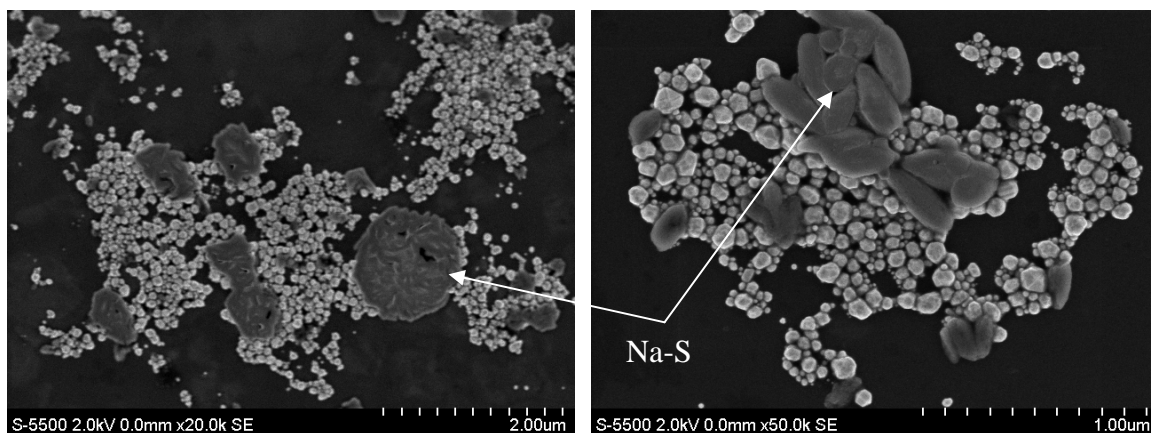


Figure 11. Secondary electron images of gold nanospheres (bright particles) and their association with Na-S material (dark material).

Oxidized Soot

The oxidized soot black dry powder sample was prepared for EM analysis by first dispersing a portion of the material in filtered ethanol using ultrasonic agitation. A lacy Formvar/carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes.

The sample was comprised of aciniform structures consisting of agglomerated rounded and irregularly shaped primary particles. Primary particles were not

well defined but appeared to be uniform in size averaging 20-40 nm in diameter. Agglomerates were usually observed on the order of micrometers. The EDS analysis of the material confirmed that the particles were composed of carbon. EM micrographs and an elemental spectrum of aciniform structures are presented in Figure 12. Figures 13 through 15 illustrate the primary particles at an increased magnification. The microstructure of the material as shown in Figure 15 shows no concentric layering in the primary particles.

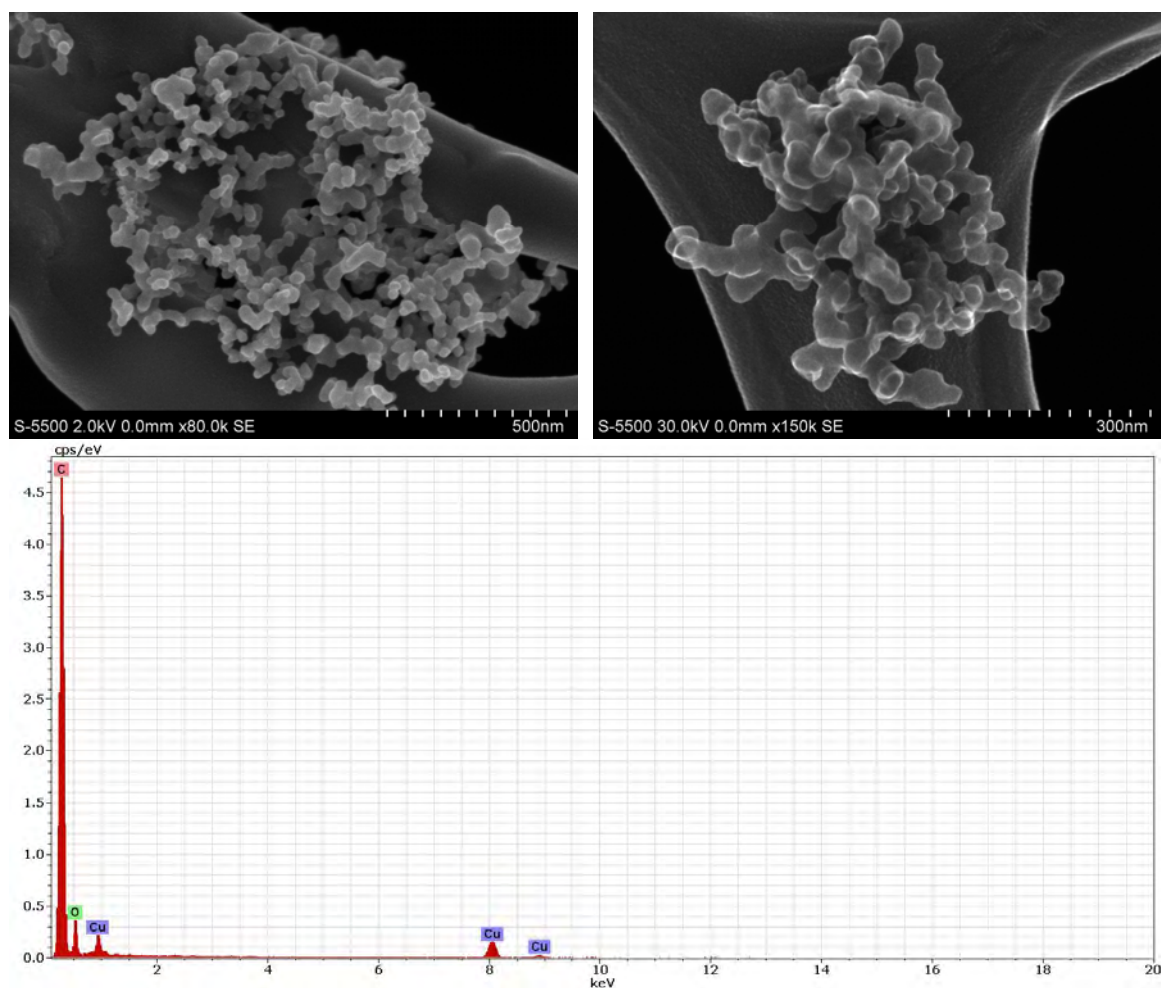


Figure 12. Secondary electron images and elemental spectrum of oxidized soot.

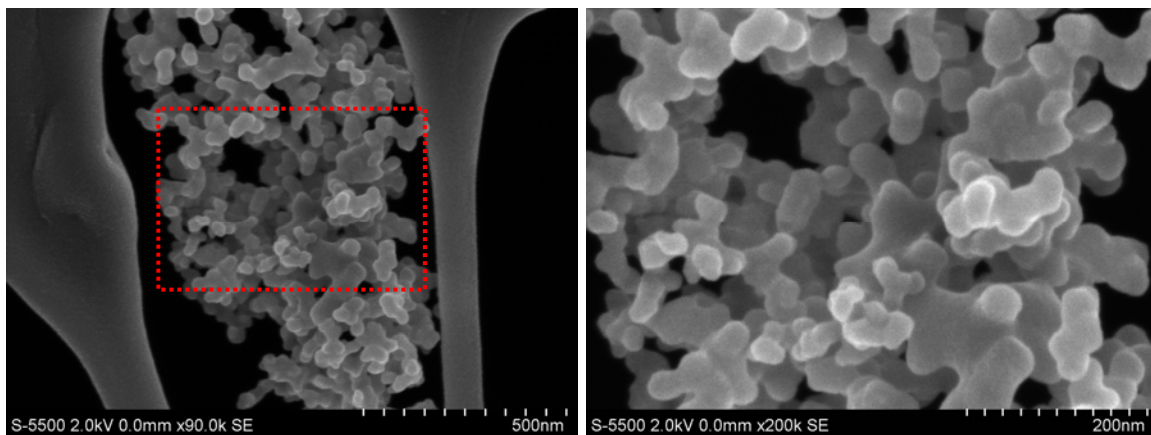


Figure 13. Secondary electron images of oxidized soot.

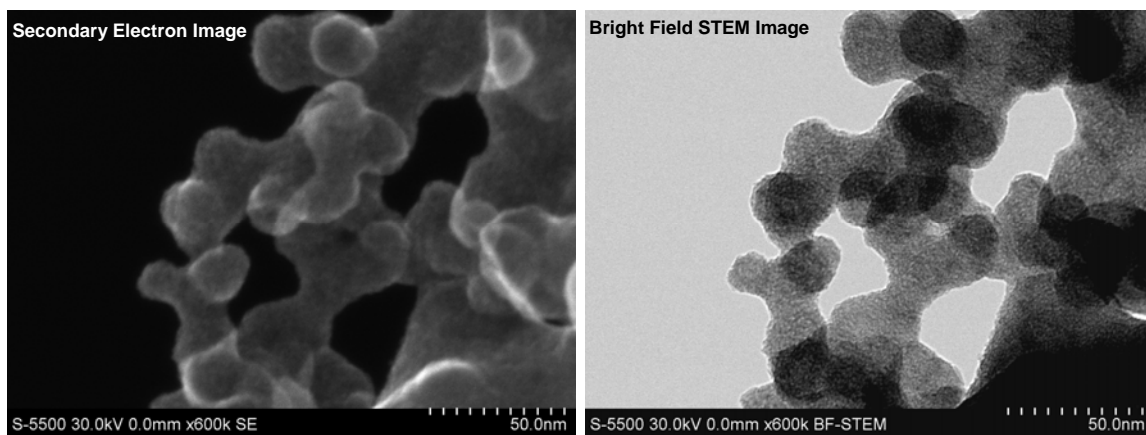
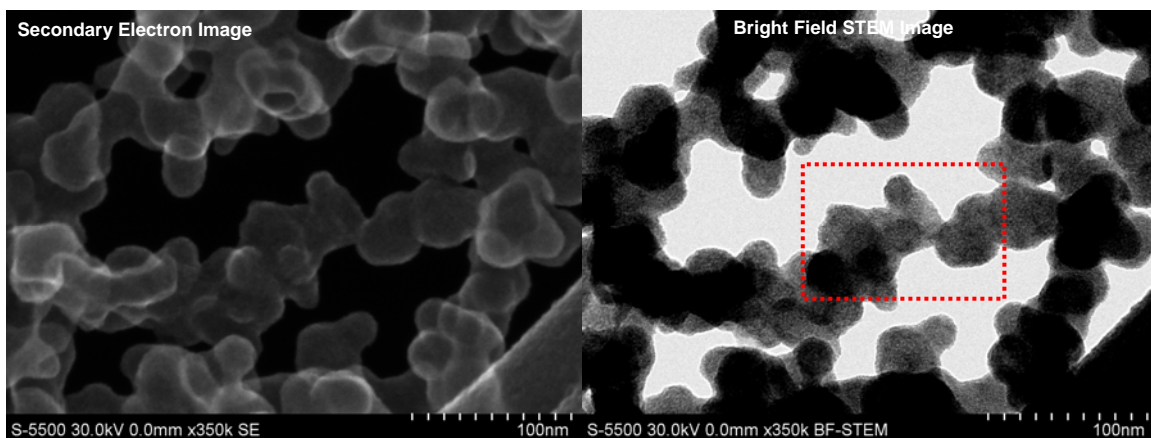


Figure 14. Secondary electron and bright field STEM images of oxidized soot.



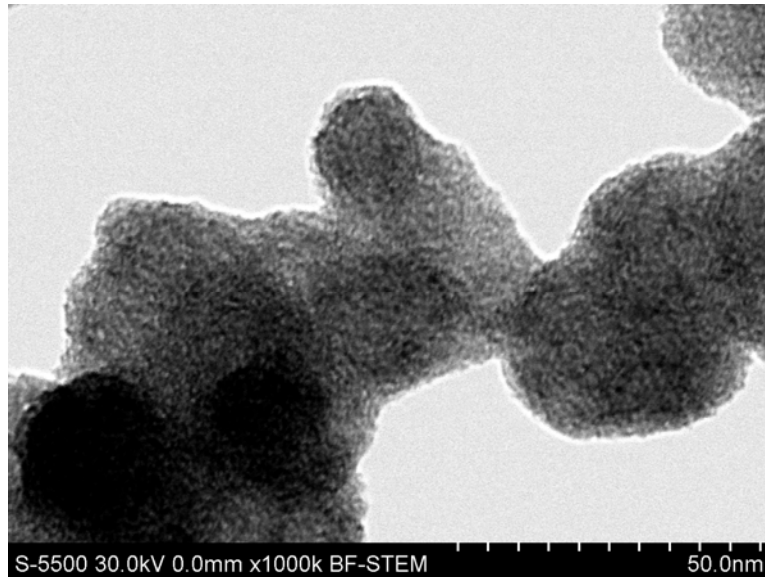


Figure 15. Secondary electron and bright field STEM images of oxidized soot.

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted.

Should you have any questions regarding this information, please do not hesitate to contact us.

Sincerely,

A handwritten signature in cursive script that reads "Kristin L. Bunker".

Kristin L. Bunker, Ph.D.
Senior Scientist

cc: G. Casuccio (RJLG)

Appendix C

Photos of Fumed Silica Process in John Kerr's Lab 62-246

Appendix C - Photos of Fumed Silica Process in John Kerr's Lab 62-246



Fig. C1. Fume hood in 62-246



Fig. C2. John Kerr works with fumed silica in 62-246 fume hood



Fig C3. Fume hood exhaust ducts in 62-246



Fig. C4. John Kerr works with fumed silica in 62-246 fume hood



Fig. C5. Fumed silica used in 62-246

Appendix C - Photos of Fumed Silica Process in John Kerr's Lab 62-246



Fig. C5. John Kerr uses glove box in 62-246

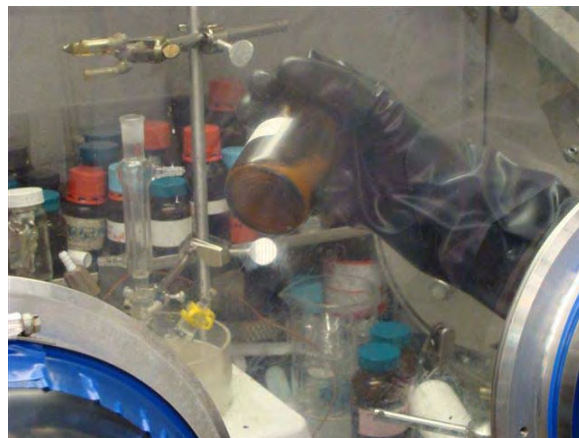


Fig. C7. John Kerr uses glove box in 62-246

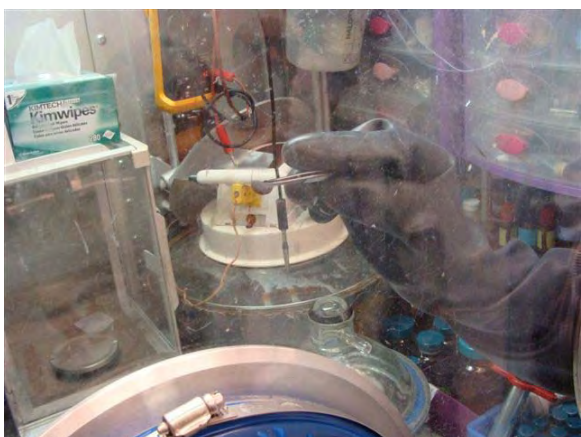


Fig. C8. John Kerr uses glove box in 62-246



Fig. C9. Posting outside entry to 62-246



Fig. C9. John Kerr uses glove box in 62-246



Fig. C10. Inert atmosphere glove box in 62-246

Appendix D

Photos of Work with Graphene/Gold in
Tom Richardson's Lab, 62-342

Appendix D - Photos of Work with Graphene/Gold in Tom Richardson's Lab, 62-342



Fig. D1. Postings at entrance to 62-342

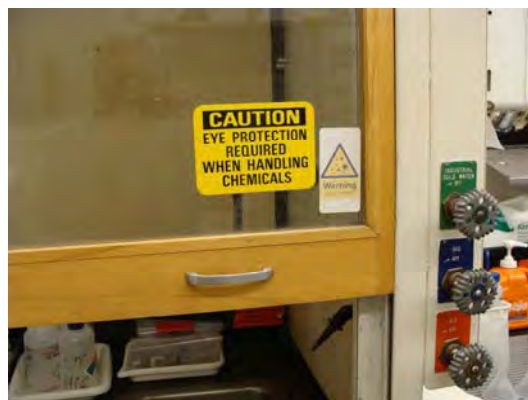


Fig. D2. Fume hood in 62-342



Fig. D3. KJ Jeon works with graphene/gold in fume hood in 62-342



Fig. D4. Nanomaterials used in fume hood in 62-342



Fig. D5. KJ Jeon works with graphene/gold in fume hood in 62-342



Fig. D6. KJ Jeon works with graphene/gold in fume hood in 62-342



Fig. D7. KJ Jeon works with graphene/gold in fume hood in 62-342

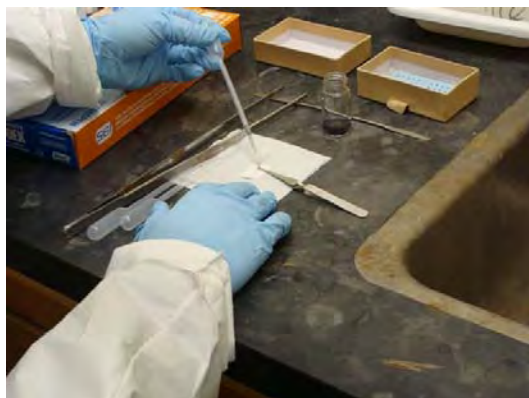


Fig. D8. KJ Jeon works with graphene/gold in fume hood in 62-342



Fig. D9. Work area in fume hood in 62-342



Fig. D10. Condensation particle counter in fume hood in 62-342



Fig. D11. Electron microscopy grid in fume hood in 62-342

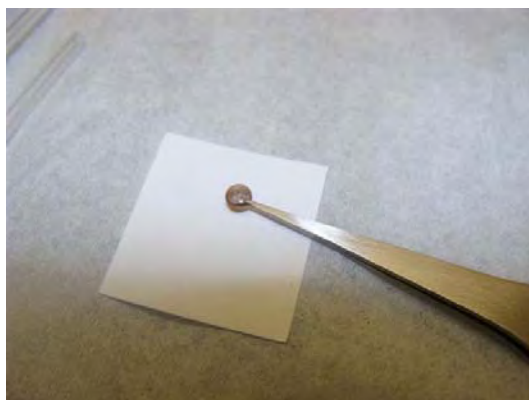


Fig. D12. Electron microscopy grid in fume hood in 62-342

Appendix E

Photos of Electroactive Polymer Research in Tom Richardson's Lab, 62-342

Appendix E - Photos of Electroactive Polymer Research in
Tom Richardson's Lab, 62-342



Fig. E1. Condensation particle counter in 62-342

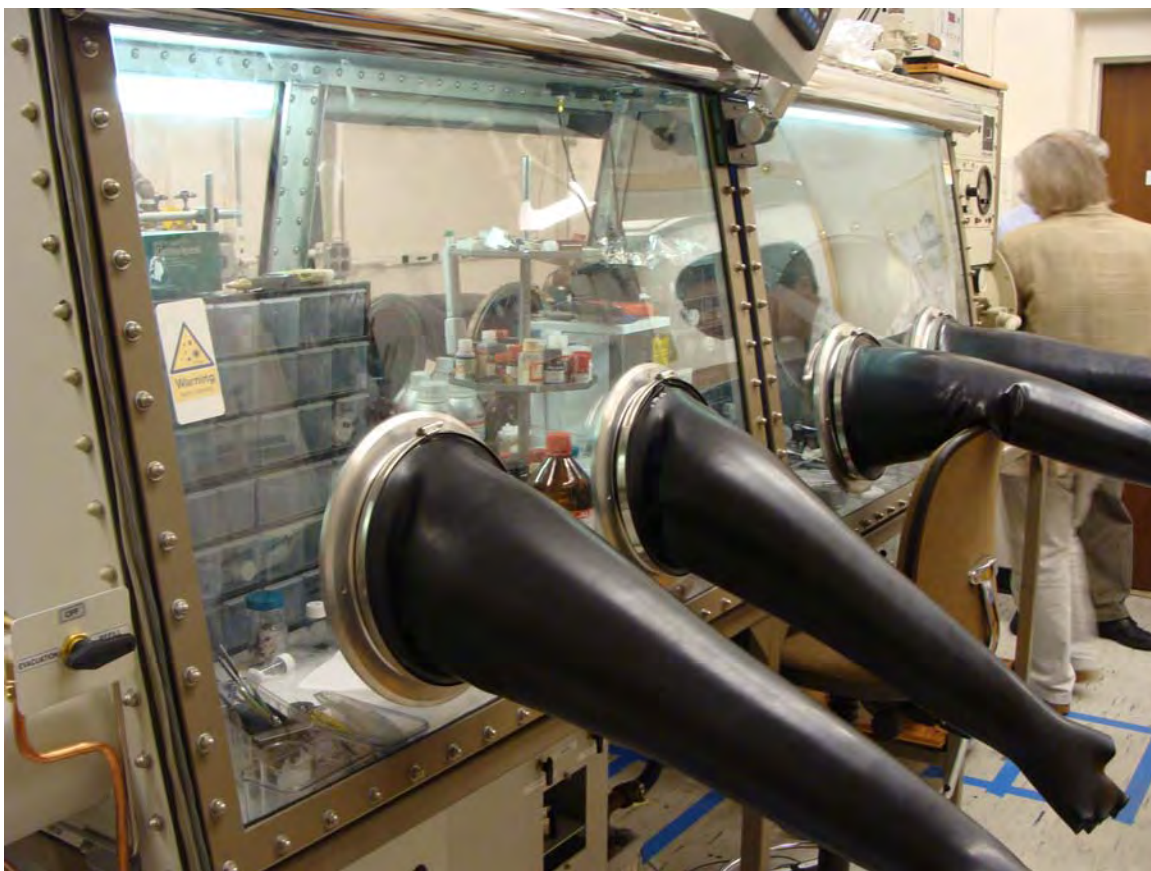


Fig. E2. Inert glove box in 62-342

Appendix E - Photos of Electroactive Polymer Research in
Tom Richardson's Lab, 62-342



Fig. E3. Tom Richardson works in glove box in 62-342; Linnea Wahl and Randy Ogle observe



Fig. E4. Tom Richardson works with acetylene black in glove box in 62-342



Fig. E5. Tom Richardson works with acetylene black in glove box in 62-342



Fig. E6. Acetylene black on scale in glove box in 62-342

Appendix E - Photos of Electroactive Polymer Research in
Tom Richardson's Lab, 62-342



Fig. E7. Work with acetylene black in glove box in 62-342



Fig. E8. Tom Richardson works with acetylene black in glove box in 62-342



Fig. E9. Tom Richardson works with acetylene black in glove box in 62-342



Fig. E10. Tom Richardson works with acetylene black in glove box in 62-342

Appendix F

Photos of Work with Carbon Black and Lithium Compounds in Vincent Battaglia's Lab, 70-299

Appendix F - Photos of Work with Carbon Black and Lithium Compounds in
Vincent Battaglia's Lab, 70-299



Fig. F1. Nanomaterial storage in 70-299



Fig. F2. Honghe Zheng demonstrates work with carbon black and lithium compounds in 70-299 inert glove box



Fig. F3. Lithium compounds in 70-299 inert glove box



Fig. F4. Carbon black in 70-299 inert glove box



Fig. F5. Honghe Zheng demonstrates work with carbon black and lithium compounds in 70-299 inert glove box; Linnea Wahl observes

Appendix F - Photos of Work with Carbon Black and Lithium Compounds in
Vincent Battaglia's Lab, 70-299



Fig. F6. Pass-through chamber of inert glove box in 70-299



Fig. F7. Condensation particle counter on table top in 70-299

Appendix G

Photos of Work with Silicon Powder in Vincent Battaglia's Lab, 70-299

Appendix G - Photos of Work with Silicon Powder in Vincent Battaglia's Lab, 70-299



Fig. G1. Silicon nanopowder stored in inert glove box in 70-299



Fig. G2. Fume hood postings in 70-299



Fig. G3. Fume hood in 70-299



Fig. G4. Xiangyun Song demonstrates work with silicon nanopowder in fume hood in 70-299



Fig. G5. Work with silicon nanopowder in fume hood in 70-299



Fig. G6. Xiangyun Song demonstrates work with silicon nanopowder in fume hood in 70-299

Appendix H

Photos of Work with Nanopowder in Vincent Battaglia's Lab, 70-299



Fig. H1. Demonstration of work with nanopowders if fume hood in 70-299



Fig. H2. Demonstration of work with nanopowders if fume hood in 70-299

Appendix I

Photos of Nanopowder Analysis in Vincent Battaglia's Lab, 70-206



Fig. I1. Xiangyun Song demonstrates use of Micromeritics Model Tristar 3000 in 70-206



Fig. I2. Xiangyun Song demonstrates use of Micromeritics Model Tristar 3000 in 70-206

Appendix J

Photos of Carbon Nanotube Storage in Gao Liu's Lab, 70-226



Fig. J1. Carbon nanotube storage in dessicator in 70-226



Fig. J2. Carbon nanotube storage in dessicator in 70-226

Appendix K

Photos of Graphene Research in Robert Kostecky's Lab, 70-108

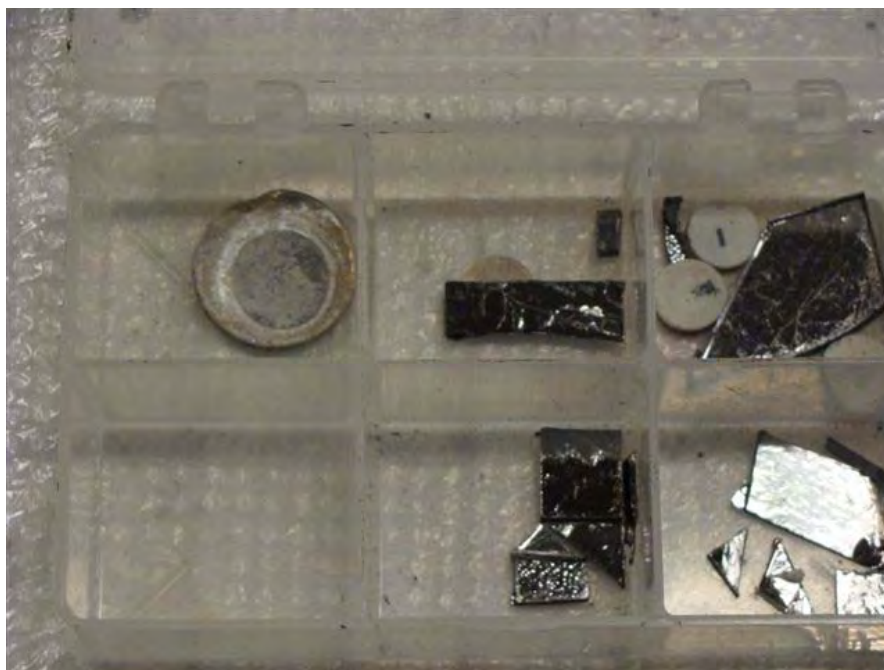


Fig. K1. Starting material for graphene preparation in 70-108



Fig. K2. Starting material for graphene preparation in 70-108

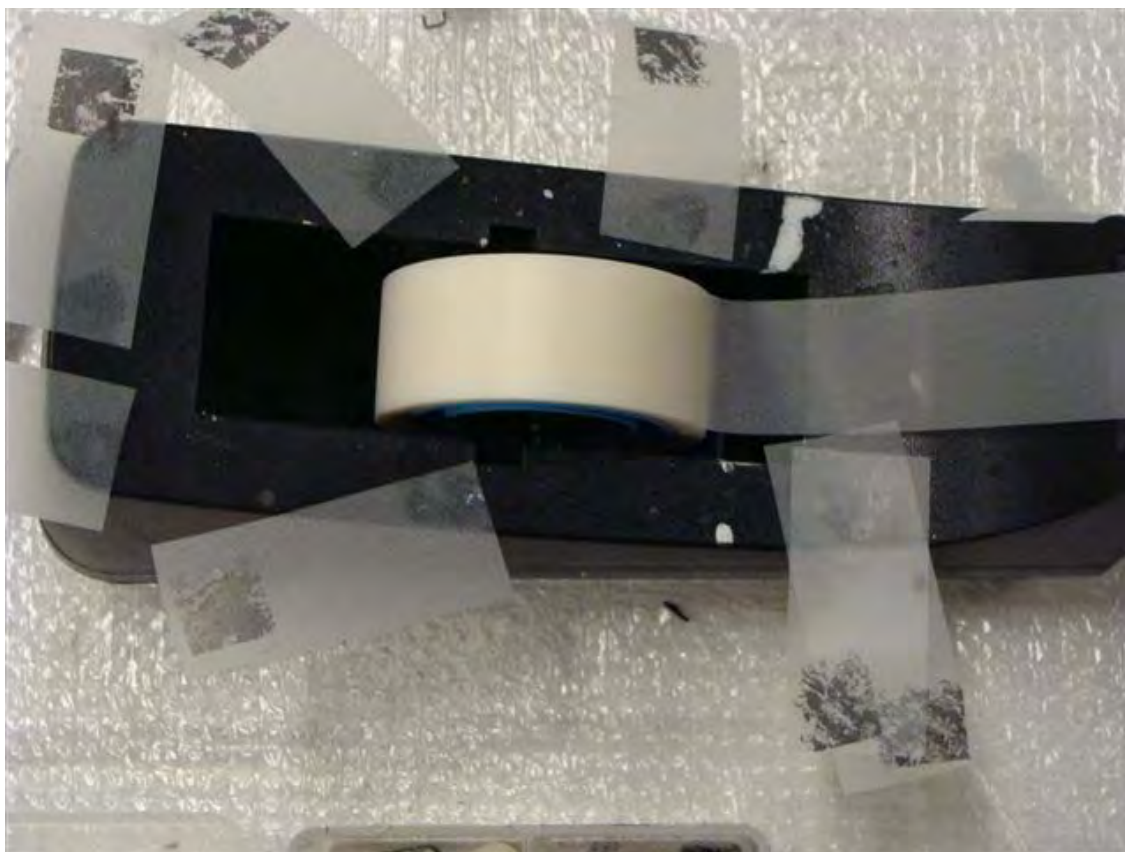


Fig. K3. Graphene prepared using transparent tape method in 70-108

Appendix L

Photos of Laser Ablation Process in Samuel Mao's Lab, 70-163

Appendix L - Photos of Laser Ablation Process in Samuel Mao's Lab, 70-163



Fig. L1. Posting on door to 70-163



Fig. L2. Local ventilation in 70-163



Fig. L3. Zhixun Ma discusses process leading to vapor condensate production in 70-163

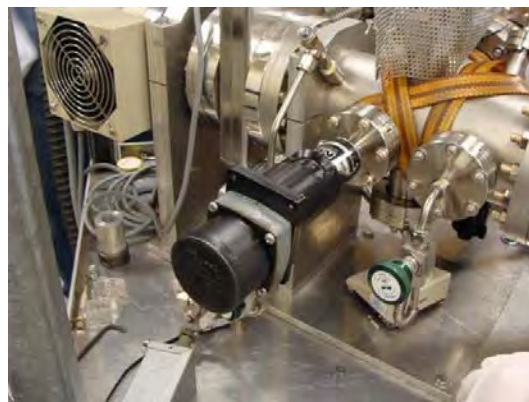


Fig. L4. Laser ablation equipment in 70-163

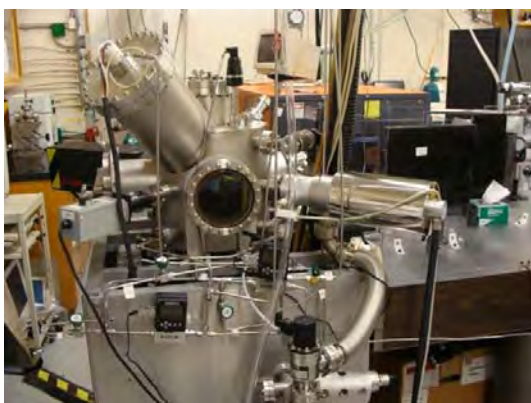


Fig. L5. Laser ablation equipment in 70-163

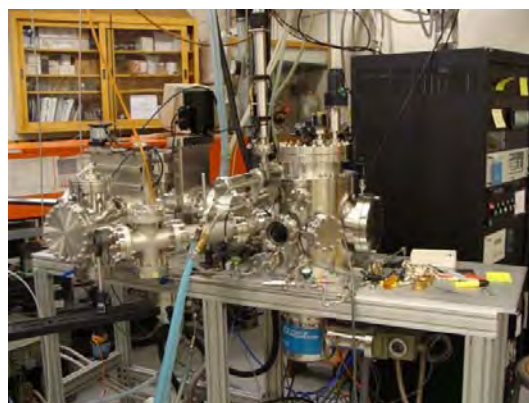


Fig. L6. Laser ablation equipment in 70-163

Appendix L - Photos of Laser Ablation Process in Samuel Mao's Lab, 70-163



Fig. L7. Laser ablation equipment in 70-163



Fig. L8. Zhixun Ma discusses process leading to vapor condensate production in 70-163

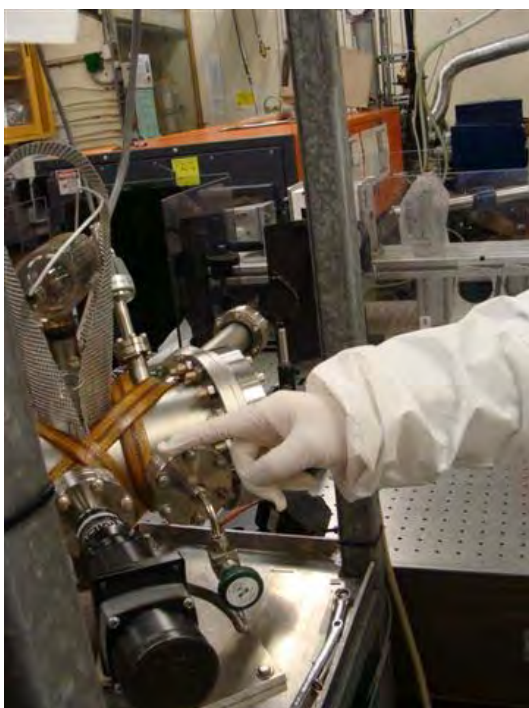


Fig. L9. Zhixun Ma discusses process leading to vapor condensate production in 70-163



Fig. L10. Condensation particle counter on table top in 70-163

Appendix M

Photos of Laser Ablation Research in Rick Russo's Lab, 70-157

Appendix M - Photos of Laser Ablation Research in Rick Russo's Lab, 70-157



Fig. M1. Posting outside 70-157



Fig M2. Laser ablation apparatus in 70-157

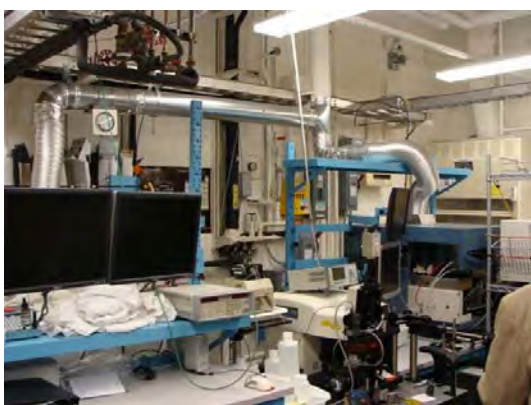


Fig M3. Laser ablation apparatus in 70-157

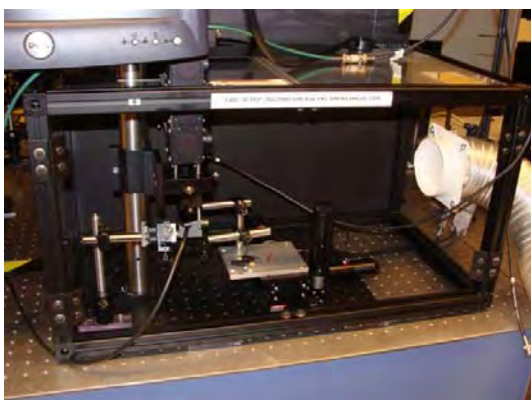


Fig M4. Laser ablation apparatus in 70-157



Fig M5. Condensation particle counter on table top in 70-157

Appendix N

Photos of Nanomaterials Research in Rick Russo's Lab, 70-157



Fig. N1. Matt Lucas discusses research process with nanomaterials in 70-157



Fig. N2. Fume hood in 70-157 where nanomaterials are used

Appendix O

Photos of Gold Rods and Spheres Research in
Don Lucas's Lab, 70-291/293

Appendix O - Photos of Gold Rods and Spheres Research in
Don Lucas's Lab, 70-291/293



Fig. O1. Posting outside 70-291/293



Fig. O2. Condensation particle counter
on table top in 70-291/293



Fig. O3. Fume hood for gold
nanorod/nanosphere work in 70-291/293



Fig O4. Jay James demonstrates gold
nanorods/nanospheres use in 70-291/293



Fig. O5. Jay James demonstrates gold
nanorods/nanospheres use in 70-291/293



Fig O6. Don Lucas and Jay James
discuss use of gold nanorods/
nanospheres in 70-291/293

Appendix P

Photos of Carbon Soot Research in Don Lucas's Lab, 70-291/293



Fig. P1. Don Lucas demonstrates work with carbon soot in 70-291/293; Linnea Wahl observes



Fig. P2. Don Lucas (R) discusses work with carbon soot with Randy Ogle (L) in 70-291/293

Appendix Q

Photos of Sputter Deposition Research in Andre Anders' Lab, 70-274

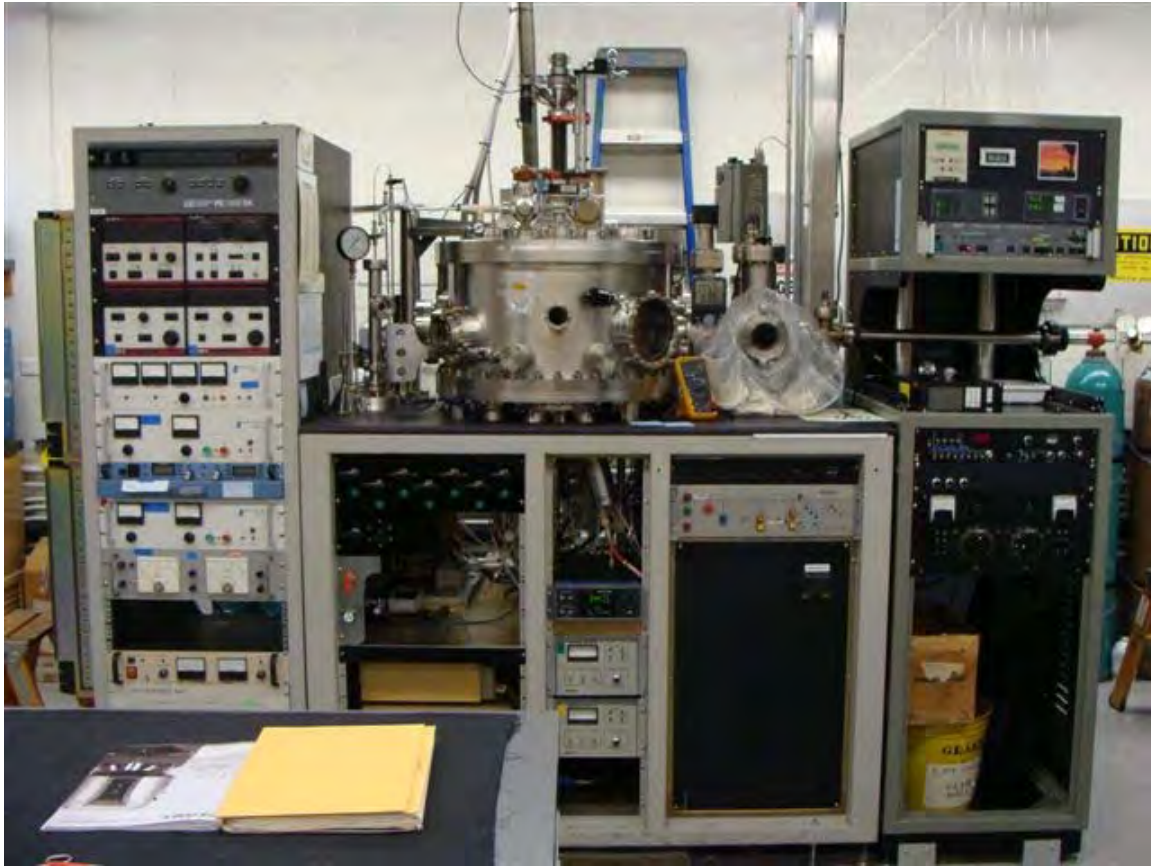


Fig. Q-1. Sputter deposition apparatus in 70-274